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NITROGEN TRIFLUORIDE (NF_3) OXIDIZER SYSTEMS DESIGN CRITERIA

PHASE II - TECHNICAL REPORT

AEROJET LIQUID ROCKET COMPANY
SACRAMENTO, CALIFORNIA 95813

Phase I
B014 992

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NOTICE

FOREWORD

This report covers the work performed under Contract F04611-76-C-0058, "Nitrogen Trifluoride (NF_3) Oxidizer Systems Design Criteria," performed by the Aerojet Liquid Rocket Company at Sacramento, California 95813 for the Air Force Rocket Propulsion Laboratory, Edwards, California 93523. The performance period covered from 1 August 1976 to 30 November 1977 and documents the work conducted as Phase II of the contract.

The program Manager is Dr. S. D. Rosenberg; the project manager and principal investigator is Dr. E. M. Vander Wall. The work conducted in Phase II - Compatibility Determinations was performed primarily by R. L. Beegle, Jr., Senior Chemist, J. A. Cabeal, Associate Chemist, R. K. Schaplowsky, Associate Chemist, T. A. Freitag, Engineer and R. E. Anderson, Chemistry Specialist. Technical advice was supplied by G. R. Janser, Engineering Specialist for Metals, and J. J. Shore, Engineering Specialist for non-metallic materials.

The program was adminstered under the direction of the Air Force Rocket Propulsion Laboratory, Lt. William T. Leyden III, Project Manager.

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The chemical compatibility of nitrogen trifluoride was experimentally determined with 29 metallic materials and 22 non-metallic materials. Twelve types of tests were used in the determination; cleaning and passivation evaluation, static exposure tests, fracture mechanics/toughness tests, gaseous flow at high temperatures, adiabatic compression tests, mechanical impact test, liquid flow impact tests, screening tests, disposal tests, nitrogen trifluoride chemical analyses, water-hammer tests and passivation (continued on reverse)

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trifluoride chemical analyses, water-hammer, contaminant effects on compatibility, impurity effects on corrosion, gaseous corrosion under flow conditions, compressibility factors, screening tests, entropy of NF_3.

20. (Cont'd)

film evaluations. Five common contaminants were included in the tests in order to determine their effect on chemical compatibility. Static exposure conditions ranged in temperature from 195 to 344 K and in pressure from 3.45 to 17.24 MN/m^2.

sq m

None of the metals tested exhibited corrosion penetration rates of greater than 1 mil per year during a 270 day exposure period when exposed to the NF_3; however the presence of HF or H_2O enhanced the corrosion rates significantly. Of the non-metals investigated, the fluorocarbons exhibited the best compatibility with NF_3.

Under load-conditions several of the metals were found to be susceptible to stress corrosion cracking. Under dynamic conditions, nickel, Inconels, and the 300 series stainless steels were found to be suitable metals for use with NF_3.

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LIST OF FIGURES (cont.)

1.0 INTRODUCTION

The objective of the "Nitrogen Trifluoride (NF_3) Oxidizer Systems Design Criteria" program, Contract F04611-76-C-0058, is to obtain compatibility and safety data and to prepare a comprehensive Design Criteria Handbook for NF_3 reactant systems. To attain the program objective, this program is conducted in three phases. Phase I consisted of a literature search and data assessment; Phase II consisted of experimental compatibility determinations; and Phase III consists of formulating information obtained from Phases I and II into a nitrogen trifluoride design criteria handbook. The work conducted as Phase I of the program was incorporated in the report AFRPL-TR-76-75 "Nitrogen Trifluoride (NF_3) Oxidizer Systems Design Criteria", Phase I - Technical Report, Aerojet Liquid Rocket Company, Sacramento, CA 95813 (September 1976). The work conducted as Phase II of the program is the subject of this report. The work conducted as Phase III of the program will be incorporated in USAF Propellant Handbooks AFRPL-TR-77-71, "Nitrogen Trifluoride Volume III, Part A, Systems Design Criteria" Aerojet Liquid Rocket Company, Sacramento, CA and in AFRPL-TR-77-72 "Nitrogen Trifluoride, Volume III, Part B, Bibliography" Aerojet Liquid Rocket Company, Sacramento, CA.

This report is organized in the following manner: (1) Introduction, (2) Experimental Results and Discussion, and (3) Conclusions.

2.0 EXPERIMENTAL RESULTS AND DISCUSSION

The objective of this program is to obtain compatibility and safety data for nitrogen trifluoride usage and to prepare a comprehensive Design Criteria Handbook for nitrogen trifluoride reactant systems. The work conducted in Phase II of the program involves experimental compatibility determinations which are documented in the report. There are twenty-nine metallic materials included in the program:

Stainless Steels 301 (Cryoformed), 303, 304, 304L, 316L, 321, 347, 17-4PH, A-286

1010-1020 Steel

OFHC copper, annealed

Aluminums 2219 T-87, 6061 T-6, 1100, 2014

Nickels 200 annealed, 270 annealed

Monel 400, annealed

Inconels 718 STA, 625

Titaniums 6Al-4V STA, 5Al-2.5 Sn ELI

CRES Nitronic 40

Maraging Steels 200 and 250

Beryllium Copper

Carpenter Custom 455

Aluminum Bronze 623

Tungsten

Narloy A

There are twenty-two non-metallic materials included in the program:

Viton	Fluorosilicone elastomer Silastic LS53
Polytetrafluoroethylene	Polyethylene
FEP Teflon	Polypropylene
PFA Teflon	Kevlar
Kel-F 81 CTFE	Carbon (CDJ-83)
Rulon (CaF_2-Filled)	Carbon (CJPS)
Neoprene	Krytox
Tygon	Vacuum Stripped Krytox (3L-38RP)
Mylar	Fluorosilicone (FS 3451)
Lucite	Dry Powder TFE (MS-122)
Epoxy (EA-934)	Kalrez (Dupont ECD-006)

2.0, Experimental Results and Discussion (cont.)

There are five material contaminants included in the program:

Fingerprints
Petroleum Jelly
Light weight machine oil
Brazing Flux
Fluorocarbon Oil (FC-75)

Twelve types of tests were used to obtain the necessary design criteria data. They are:

Cleaning and Passivation
Static Tests
Fracture Mechanics/Toughness Tests
Flow Tests
Adiabatic Compression Tests
Mechanical Impact Tests
Flow Impact Tests
Screening Tests
Disposal Tests
Propellant Analyses
Water Hammer Tests
Passivation Film Evaluation Tests

This section of the report is presented in the following order: (1) Cleaning and Passivation Pretreatment of Materials, (2) Static Tests, (3) Fracture Mechanics/Toughness Tests, (4) Gaseous Flow Tests, (5) Adiabatic Compression Tests, (6) Mechanical Impact Tests, (7) Flow Impact Tests, (8) Waste Disposal Tests, (9) Nitrogen Trifluoride Analyses and Compressibility Factors, (10) Water Hammer Tests, (11) Nature and Rate of Formation of Passivation Films, (12) Solubility of Passivation Films in Liquid NF_3, (13) Effect of Contaminants on Metals, (14) Effect of Impurities on NF_3 Compatibility, and (15) Gaseous Corrosion Rates of Metals Under Flow Conditions.

2.0,

2.1 CLEANING AND PASSIVATION PRETREATMENT OF MATERIALS

The objective of this task was to establish at the outset of the experimental program the proper procedures for cleaning and passivating materials prior to testing, to verify the validity of the test procedures, and to identify any gross incompatibilities which might exist between nitrogen trifluoride and the selected materials.

2.1.1 Cleaning and Passivation Pretreatment of Metals

The purpose of this task was to establish early in the experimental program the appropriate procedures for preparation of the metal surfaces prior to exposure to nitrogen trifluoride for prolonged periods of time. The two factors which required investigation were:

(1) The effect of cleaning and pickling on metal/nitrogen trifluoride compatibility and

(2) The effect of pre-exposure to nitrogen trifluoride as a passivation step and whether this type of "passivation" is necessary to establish chemical compatibility between nitrogen trifluoride and metals.

2.1.1.1 Cleaning Procedures for Metals

The metal specimens used for testing were cleaned according to two procedures which are as follows. The first procedure consists of a detergent wash using Turco Plaudit as the detergent. The washing is followed by degreasing in an isopropanol bath, a deionized water rinse, an additional rinse with isopropanol and then vacuum drying the specimens for 4 hours at 333K (140F). The 1010 steel specimens were placed immediately in a vacuum flask after the last isopropanol rinse to minimize oxidation of the samples. The second procedure involves all the steps of the first procedure plus immersing the specimens in appropriate pickling solutions, followed by a deionized water rinse, then an isopropanol rinse and a final drying under vacuum at 333K (140F).

The pickling solutions are defined for the various materials in Table 2.1-1. Because the Monel 400, Nickel 200, and Nickel 270 appeared to have traces of copper on the specimens after pickling in the specified solution, the specimens were momentarily dipped in the pickling solution used for the 300 series stainless steels to remove the trace of copper. The pickling solution recommended by the manufacturer for the maraging steels was totally inadequate. Its use produced a tenacious smut on the metal specimens which was removed by hydrohoning with glass spheres.

TABLE 2.1-1

PICKLING SOLUTIONS USED FOR VARIOUS METALLIC SPECIMENS

Materials	Pickling Solutions and Procedure
300 Series Stainless Steel, 17-4 PH, Nitronic-40 A-286, Carpenter Custom 455	10% HNO_3, 4% NF, 86% H_2O, Immersed Specimens for 5 Minutes @ 110°F
2219 Aluminum 6061 Aluminum 1100 Aluminum 2014 Aluminum	3.5% of 85% H_3PO_4, 2 gr$Na_2Cr_2O_7 \cdot 2H_2O$ per 100 ML Sol'n 96.5% H_2O Immersed Specimens for 5 Minutes @ 212°F
Inconel 625 Inconel 718	10% HNO_3, 5% HF, 85% H_2O Immersed Specimens for 5 Minutes @ 110°F
Monel 400 Nickel 200 Nickel 270	50% HCl (20°Bé) 3g $CuCl_2$ per 100 ML of Sol'n 50% H_2O Immersed Specimens for 5 Minutes @ 180°F
6Al-4V Titanium 5Al-2.5 Sn Titanium	20% HNO_3, 1% HF, 79% H_2O Immersed 6Al-4VTi for 15 Minutes @ 120°F Immersed 5Al-2.5 Sn Ti for 5 Minutes @ 120°F
1010 Steel	8% H_2SO_4 (66° Bé), 3% HF, 89% H_2O Immersed Specimens for 5 Minutes @ 120°F
OFHC Copper Beryllium	75% HCl (20° Bé), 25% H_2O Immersed Specimens for 2 Minutes at Room Temperature
Maraging Steels	18% H_2SO_4, 82% H_2O at 150-160°F Immerse Specimens Until Clean
Aluminum Bronze	4-15% H_2SO_4 (1.83 SP GR) by Volume Remainder H_2O, Immerse Specimens 1/2 to 15 Minutes at Room Temperature to 140°F

2.1.1.2 Passivation Procedures for Metals

With regard to passivation, the degree of prepassivation that is required in NF_3 systems was not adequately defined. It remained to be demonstrated that self-passivation occurs on clean surfaces at appropriate reaction rates. From an operational standpoint, it is desirable that the treatment can be accomplished with the NF_3 itself. It is also recognized that such treatments are normally conducted at a temperature above the designed use temperature when it is practical to do so. Based on the foregoing, metal specimens were subjected to three conditions with regard to passivation: (1) no pretreatment, (2) exposure to NF_3 vapor at a few atmospheres pressure for at least two hours at room temperature, and (3) exposure to F_2 vapor at a few atmospheres pressure for at least two hours at room temperature.

2.1.1.3 Test Apparatus and Procedures

The metals specimens were tested in the form of coupons which were 4.45 cm (1.75 in.) long, 1.59 cm (.625 in.) wide, and the thickness varied from 0.025 to 0.318 cm (.010 to .125 in.). Two holes, 0.318 cm (.125 in.) in diameter were drilled in the coupons so that they could be held in position during exposure to the nitrogen trifluoride. The rack with the specimens is shown in Figure 2.1.1. The racks were inserted into containers which were fabricated from 5.1 cm (2 in.) diameter 304L pipe and a pipe end-cap. A stainless steel bellows valve was welded to the other end cap and this assembly was welded to the 5.1 cm (2 in.) diameter pipe after the rack was in place. An internal and external purge of argon was maintained during the final welding operation. The test container was leak-checked by pressurization with helium, then evacuated to less than 1 mm (Hg) pressure and filled through the valve with the desired gas. A photograph of a test container is shown in Figure 2.1.2.

The aluminum alloy specimens were not loaded as described above but were positioned between two slotted Teflon plugs and then inserted into a container fabricated from 1.9 cm (.75 in.) diameter 304L pipe with an end cap. The pipe was then sealed by welding on the other end cap to which a .64 cm (.25 in.) diameter 304 fill tube was attached. The welding was conducted as described for the 5.1 cm (2 in.) diameter containers. After leak checking and subjecting the contents to the desired pretreatment, the tubes were filled with the desired quantity of nitrogen trifluoride, by condensation in liquid nitrogen. While the contents were immersed in the liquid nitrogen, the fill tube was crimped and then welded. The container is shown in Figure 2.1.3.

The test matrix for screening the metal/nitrogen trifluoride interactions is presented in Table 2.1-2. The F_2 passivation

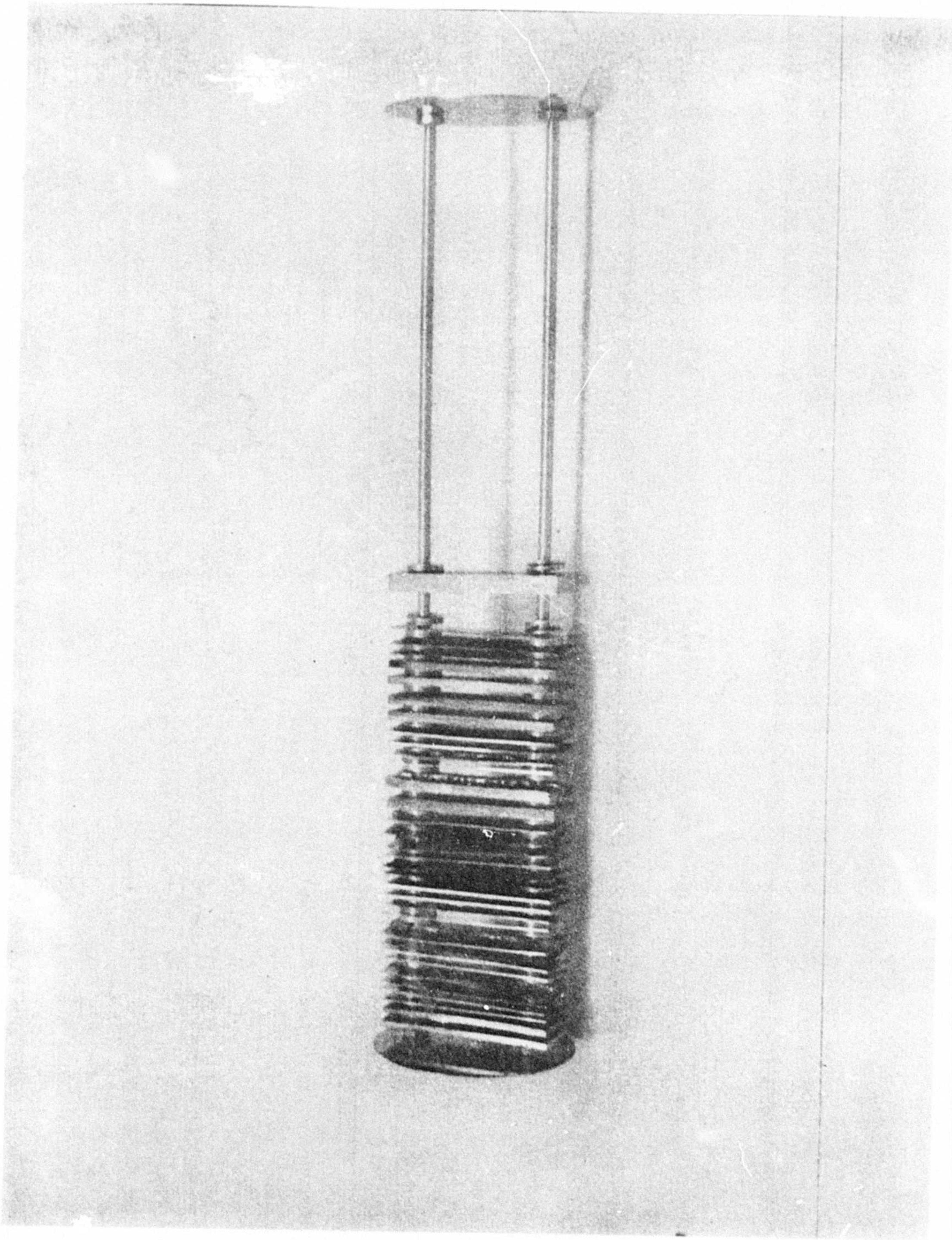


Figure 2.1.1. Metal Specimens Mounted on a Rack for Static Testing in Nitrogen Trifluoride

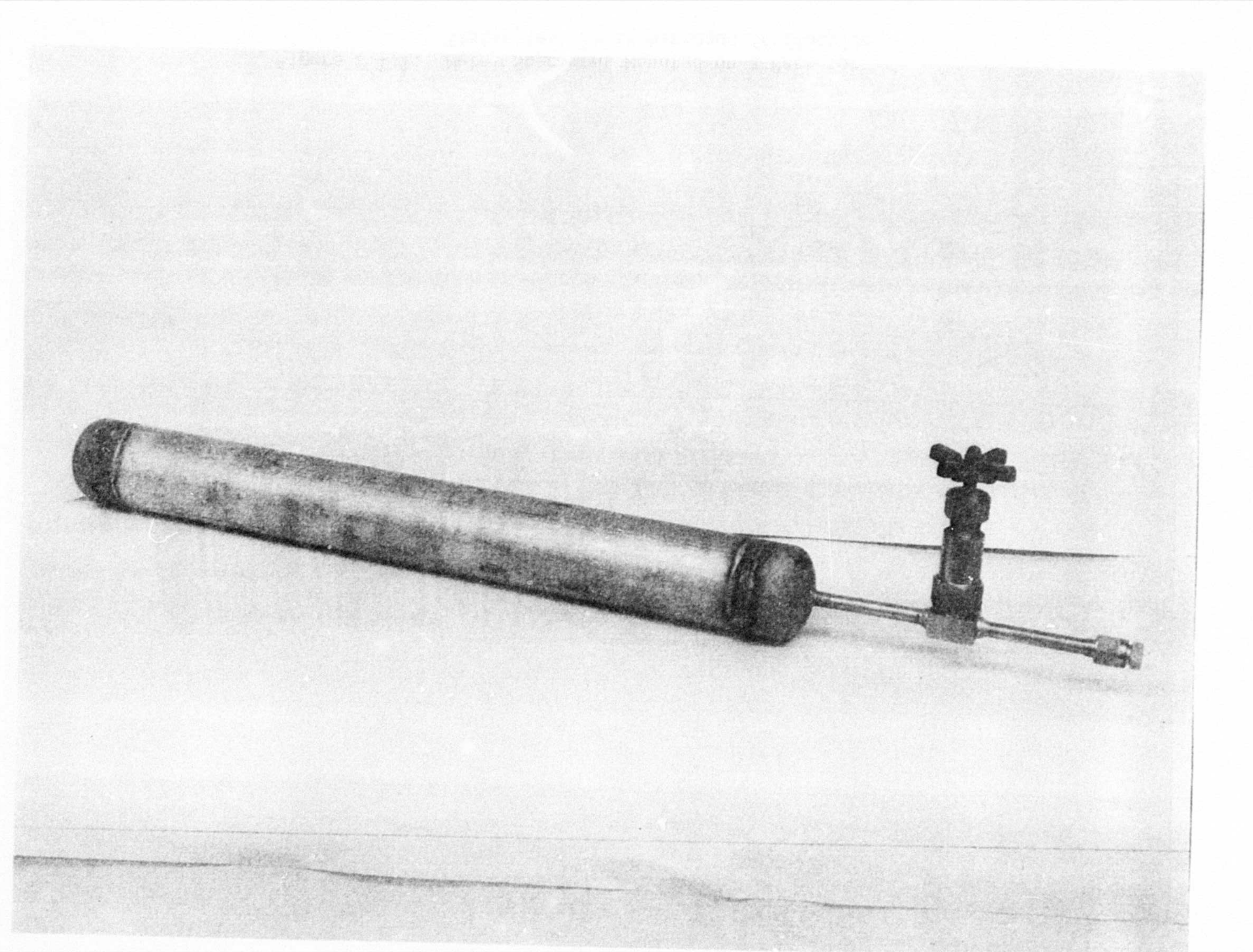


Figure 2.1.2. Container Used for Exposure of Metal Specimens Mounted on a Rack to Nitrogen Trifluoride

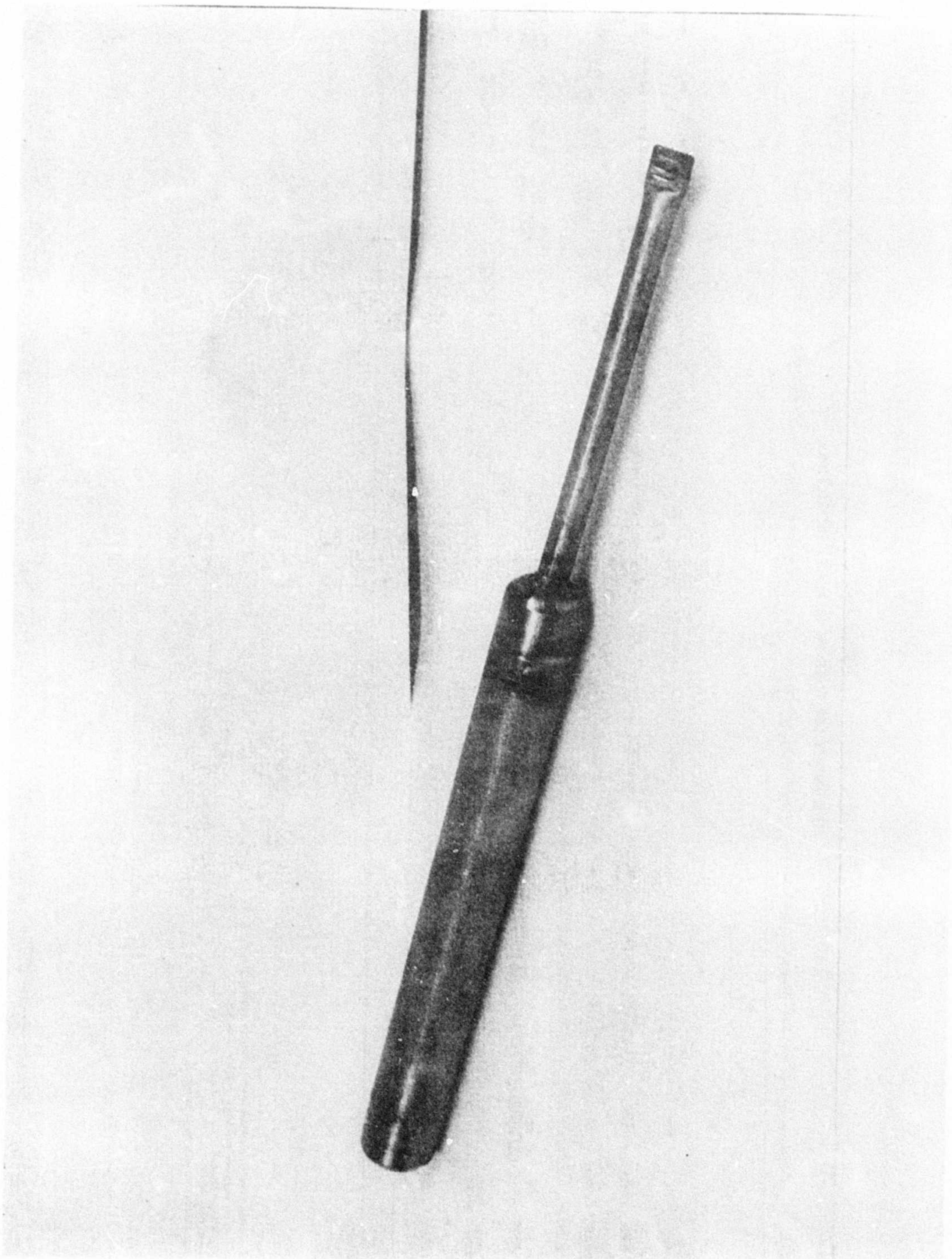


Figure 2.1.3. Container Used for Exposure of Non-Metal Specimens to Nitrogen Trifluoride

TABLE 2.1-2

TEST MATRIX FOR SCREENING METAL/NF_3 INTERACTIONS

Test Apparatus Identification Number

Test Conditions	1	2	3	4	5	6	7	8	9	10	11	12
Cleaning Procedure	R	R	R	R	R	R	R	R	R	M	M	M
Passivation Procedure	NF_3	NF_3	NF_3	None	None	None	F_2	F_2	F_2	None	None	None
Exposure Temperature	223K	223K	223K	223K	223K	223K	223K	223K	223K	223K	223K	223K
Type of Exposure	ℓ/v,v	ℓ/v,v	ℓ/v,v	ℓ/v	ℓ/v	ℓ/v	ℓ/v	ℓ/v	ℓ/v	ℓ/v	ℓ/v	ℓ/v
Metals Present	All but aluminum	304 and aluminum	304	All candidate classes but aluminum	304 and aluminum	304	All candidate classes but aluminum	304 and aluminum	304	All candidate classes but aluminum	304 and aluminum	304
Metal Condition	Parent, welded	Parent, welded	Parent, welded	Parent	Parent	Parent, welded	Parent	Parent	Parent, welded	Parent	Parent	Parent, welded
Analyses NF_3: Composition,	+	+	+	+	+	+	+	+	+	+	+	+
Dissolved Solids,	+	+	+	+	+	+	+	+	+	+	+	+
Insolubles	+	+	+	+	+	+	+	+	+	+	+	+
Metals: Changes in physical properties and appearance	+	+	+	+	+	+	+	+	+	+	+	+

Test Conditions	13	14	15	16	17	18	19	20	21	22	23	24
Cleaning Procedure	M	M	M	R	R	R	R	R	R	R	R	R
Passivation Procedure	NF_3	NF_3	NF_3	NF_3	NF_3	NF_3	NF_3	NF_3	NF_3	None	None	None
Exposure Temperature	223K	223K	223K	344K	344K	344K	344 K	344 K	344 K	344 K	344 K	344 K
Type of Exposure	ℓ/v	ℓ/v	ℓ/v	$NF_{3(g)}$ 500 psi	$NF_{3(g)}$ ∿500 psi	$NF_{3(g)}$ ∿500 psi	$NF_{3(g)}$ ∿500 psi H_2O ℓ/v	$NF_{3(g)}$ ∿500 psi H_2O ℓ/v	$NF_{3(g)}$ ∿500 psi H_2O ℓ/v	H_2O ℓ/v	H_2O ℓ/v	H_2O ℓ/v
Metals Present	All candidate classes but aluminum	304 and aluminum	304	All candidate classes but aluminum	304 and aluminum	304	All candidate classes but aluminum	304 and aluminum	304	All candidate classes but aluminum	304 and aluminum	304
Metal Condition	Parent	Parent	Parent, welded	Parent	Parent	Parent, welded	Parent	Parent	Parent, welded	Parent	Parent	Parent, welded
Analyses NF_3: Composition,	+	+	+	+	+	+	+	+	+			
Dissolved Solids,	+	+	+				+	+	+	+	+	+
Insolubles	+	+	+	+	+	+	+	+	+	+	+	+
Metals: Changes in physical properties and appearance	+	+	+	+	+	+	+	+	+	+	+	+

R - indicates a rigorous cleaning procedure which includes a pickling step
M - indicates a cleaning procedure from which pickling is excluded
ℓ/v - indicates liq. d-vapor exposure
v - indicates vapor exposure only
+ - indicates that the analysis will be performed

treatment is considered to be the most rigorous possible and undesirable from an operational viewpoint. Therefore it is used only in conjunction with the rigorous cleaning procedure which includes the pickling step. The "non-treatment" and NF_3 passivation are evaluated in conjunction with both the abbreviated and rigorous cleaning procedures. The NF_3 passivation treatment with rigorous cleaning procedure as appropriate with all the candidate metals is used in the program as the baseline condition in the metal screening tests at 223K (-50 C). Selected metal specimens were used in evaluation of the effectiveness of "non-passivated"-abbreviated cleaning, "non-passivated"-rigorous cleaning, NF_3 passivated - abbreviated cleaning, and F_2 passivated - rigorous cleaning procedures. The selected metal specimens include a representative of each class of candidate metals: an austenitic stainless steel, 17-4 PH, an Inconel, Monel 400, Nickel 200, an aluminum alloy, Ti 6Al-4V, Cu OFHC and 1010 steel. The metal specimens in each test apparatus during the 30-day exposure were grouped in accordance with their pre-test treatments in order to eliminate any cross-contamination. Control tests were conducted with 304 specimens in 304L containers to verify that metal/metal interactions did not occur in the gauged metal specimen tests.

The majority of the tests were conducted at 223 K (-50 C) in order to evaluate the material compatibility under a "worst case" condition in the liquid phase, the nitrogen trifluoride critical temperature is 233 K and the vapor pressure of nitrogen trifluoride at 223 K is approximately 3.45 MN/m^2 (500 psia). To assess the effect of temperature on the material compatibility in the vapor phase, one series of tests was included in the matrix at conditions of 3.45 MN/m^2 (500 psia) and 344 K (160 F)

The 223 K (-50 C) temperature environment was achieved by injecting liquid nitrogen into an insulated box which was fitted with an internal circulating fan. A helium-gas thermometer in connection with a U-tube containing mercury which opened and closed contacts of an electrical relay was used to operate a solenoid valve which controlled the coolant flow. The temperature was maintained within $\pm$ 3 K of the desired temperature. The 344 K (160 F) temperature environment was achieved by use of a circulating air oven which was controlled to within $\pm$ 1 K.

2.1.1.4 Test Results

The tests reported in this section were all conducted with the same cylinder of NF_3 and its analysis is reported in the tabulation presented in Table 2.1-13. All the tests were conducted in sample bombs fabricated from 304L stainless steel as described in the previous section.

One series of tests was designed to obtain information early in the program as to the general corrosive nature of the NF_3 towards the various metallic materials. The metal specimens used in the tests were cleaned and pickled with the appropriate solutions as shown in Table 2.1-1 and then subjected to nitrogen trifluoride vapor exposure at 2 atmospheres for at least two hours before the sample bombs were loaded with the required quantity of nitrogen trifluoride. The data which are presented include the type of specimen, its surface area, weights, calculated average corrosion penetration rates in both the S.I. units of picometers/sec (pm/sec) and English units of mils per year (mpy), and visual observations. The data from the test series are presented in Tables 2.1-3, 2.1-4, and 2.1-5. The several metallic specimens were grouped into a single test bomb for each specific exposure condition; the specimens listed in each table were in the same test bomb. The two exposure conditions indicated in Tables 2.1-3 and 2.1-4 are due to the fact that the initial exposure was at 223 K, but the bombs were stored following the initial exposure in a dry-ice environment, 195 K prior to the opening.

The significant items to note from the data in Tables 2.1-3, 2.1-4 and 2.1-5 are that (1) in general no catastrophic corrosion occurs in either the liquid/vapor or vapor only exposure at 233 K, the weight losses for the titanium alloys correspond to a corrosion rate of less than 0.2 pm/sec (0.2 mpy) in the liquid/vapor exposure and to a rate of .03 pm/sec (0.04 mpy) for the vapor exposure, the other materials exhibited rates which are an order of magnitude less; (2) in the vapor phase at 344 K (160 F) the titanium alloys exhibited a corrosion penetration rate of .16 pm/sec (0.2 mpy) and all the other materials were at least an order of magnitude less except for the 17-4 PH which exhibited a rate of .73 pm/sec (0.9 mpy). The behavior of the 17-4 PH may be an anomaly which is resolved by the long-duration static tests.

The next series of tests was designed to assess the merit of pickling the cleaned parent materials prior to exposure. The samples used in the test series were not highly oxidized in their as-received condition and were cleaned by the procedures described in Table 2.1-1. The data are reported in Tables 2.1-6 and 2.1-7. The data indicate a general trend that the weight losses were very slightly less with the materials which were subjected to the pickling procedure. A comparison of the data in Table 2.1-3 in which case the samples were cleaned, pickled and nitrogen trifluoride passivated with the data in Table 2.1-8 in which parent metal specimens were cleaned and nitrogen trifluoride passivated, but not pickled confirms the general trend of a very slight improvement in corrosion resistance. The benefits of the pickling procedures are best demonstrated for the titanium alloys in which case a significant improvement is apparent.

TABLE 2.1-3

DATA INDICATIVE OF THE COMPATIBILITY OF NITROGEN TRIFLUORIDE WITH VARIOUS METALS

EXPOSURE CONDITIONS: NF_3 Liquid/Vapor, 223 K, 26 days, 195 K, 20 days
SPECIMEN PRETREATMENTS: Cleaned, Pickled, NF_3 Passivated

Material	Specimen Type	Specimen Surface Area, Cm2	Specimen Weights, g			Penetration Rate		Observations
			Initial	Final	Loss	pm/sec	mpy	
301 SS, Cryoformed	Parent	15.59	7.9921	7.9922	(.0001)	0	0	Slight stain
301 SS, Cryoformed	Welded	15.74	8.7674	8.7680	(.0006)	0	0	Slight stain
304 L SS, Annealed	Parent	13.93	0.8527	0.8527	--	0	0	No apparent reaction
304 L SS, Annealed	Welded	13.93	1.5852	1.5850	.0002	.005	.006	No apparent reaction
316 ELC SS	Parent	14.21	2.0305	2.0305	--	0	0	Slight stain
316 ELC SS	Welded	14.21	2.7535	2.7533	.0002	.004	.005	Some stain
321 SS, Annealed	Parent	14.23	2.0208	2.0207	.0001	.002	.003	Some stain
321 SS, Annealed	Welded	14.23	3.0123	3.0117	.0006	.013	.016	Some stain
347 SS, Annealed	Parent	14.06	1.4079	1.4079	--	0	0	No apparent reaction
347 SS, Annealed	Welded	14.06	2.3938	2.3936	.0002	.005	.006	No apparent reaction
17-4 PH, H-1025	Parent	15.66	8.4761	8.4761	--	0	0	Very slight stain in vapor phase
17-4 PH, H-1025	Welded	15.66	8.8710	8.8708	.0002	.004	.005	No apparent reaction
Inconel 625, Annealed	Parent	14.57	3.9605	3.9606	(.0001)	0	0	No apparent reaction
Inconel 625, Annealed	Welded	14.57	4.3418	4.3419	(.0001)	0	0	No apparent reaction
Inconel 718, Annealed	Parent	14.06	1.4438	1.4439	(.0001)	0	0	Some stain
Inconel 718, Annealed	Welded	14.06	2.1674	2.1674	--	0	0	No apparent reaction
Monel 400, Annealed	Parent	14.21	2.2571	2.2570	.0001	.002	.002	Some stain
Monel 400, Annealed	Welded	14.21	3.8787	3.8788	(.0001)	0	0	Some stain
Nickel 200, Annealed	Parent	14.05	1.5159	1.5158	.0001	.002	.002	Some stain
Nickel 200, Annealed	Welded	14.05	2.1595	2.1593	.0002	.004	.005	Some stain
Nickel 270, Annealed	Parent	17.02	16.3524	16.3520	.0002	.003	.004	Considerable stain
Nickel 270, Annealed	Welded	17.02	17.2825	17.2822	.0003	.005	.006	Considerable stain
Titanium 6Al-4V, STA	Parent	14.72	2.4092	2.4056	.0036	.14	.17	Yellow deposit, heavier in liquid phase
Titanium 6Al-4V, STA	Welded	14.72	2.6307	2.6276	.0031	.12	.15	Yellow deposit, heavier in liquid phase
Titanium 5Al-2.5 Sn, ELI	Parent	14.68	2.3696	2.3674	.0022	.08	.10	Yellow deposit, heavier in liquid phase
Titanium 5Al-2.5 Sn, ELI	Welded	14.68	2.6883	2.6853	.0030	.11	.14	Yellow to brown deposit, heavier in liquid phase
C-1010 Steel	Parent	14.05	1.3384	1.3383	.0001	.002	.003	Considerable stain
C-1010 Steel	Welded	14.05	1.7895	1.7891	.0004	.009	.011	Considerable purple stain
Copper, OFHC	Parent	14.06	1.5542	1.5540	.0002	.004	.005	Some tarnish, heavier in vapor phase
Copper, OFHC	Welded	14.06	3.3738	3.3735	.0003	.006	.007	Considerable tarnish in vapor phase

TABLE 2.1-4

DATA INDICATIVE OF THE COMPATIBILITY OF NITROGEN TRIFLUORIDE WITH VARIOUS METALS

EXPOSURE CONDITIONS: NF_3 Vapor, 223 K, 3.45 MN/m^2 (500 psi) 26 days; 195 K, 15 days
SPECIMEN PRETREATMENTS: Cleaned, Pickled, NF_3 Passivation

Material	Specimen Type	Specimen Surface Area, Cm^2	Specimen Weights, g			Penetration Rate		Observations
			Initial	Final	Loss	pm/sec	mpy	
301 SS, Cryoformed	Parent	15.59	8.1121	8.1121	--	0	0	Very slight stain
301 SS, Cryoformed	Welded	15.74	8.8275	8.8274	.0001	.002	.003	No apparent reaction
304 L SS, Annealed	Parent	13.93	0.8337	0.8336	.0001	.002	.003	Very slight stain
304 L SS, Annealed	Welded	13.93	1.6492	1.6491	.0001	.002	.003	No apparent reaction
316 ELC SS	Parent	14.21	2.0367	2.0366	.0001	.002	.003	Very slight stain
316 ELC SS	Welded	14.21	3.0879	3.0876	.0003	.007	.009	No apparent reaction
321 SS, Annealed	Parent	14.23	2.0503	2.0502	.0001	.002	.003	Stain
321 SS, Annealed	Welded	14.23	2.7905	2.7903	.0002	.005	.006	No apparent reaction
347 SS, Annealed	Parent	14.06	1.4172	1.4171	.0001	.002	.003	No apparent reaction
347 SS, Annealed	Welded	14.06	2.5815	2.5813	.0002	.005	.006	No apparent reaction
17-4 PH, H-1025	Parent	15.66	8.4748	8.4747	.0001	.002	.003	Very slight stain
17-4 PH, H-1025	Welded	15.66	8.6527	8.6530	(.0003)	0	0	No apparent reaction
Inconel 625, Annealed	Parent	14.57	3.9567	3.9567	--	0	0	No apparent reaction
Inconel 625, Annealed	Welded	14.57	4.4271	4.4270	.0001	.002	.003	No apparent reaction
Inconel 718, Annealed	Parent	14.06	1.4486	1.4486	--	0	0	Slight stain
Inconel 718, Annealed	Welded	14.06	2.1143	2.1143	--	0	0	No apparent reaction
Monel 400, Annealed	Parent	14.21	2.2728	2.2726	.0002	.005	.006	Very slight stain
Monel 400, Annealed	Welded	14.21	4.6882	4.6880	.0002	.005	.006	No apparent reaction
Nickel 200, Annealed	Parent	14.05	1.5199	1.5198	.0001	.002	.002	No apparent reaction
Nickel 200, Annealed	Welded	14.05	2.1883	2.1881	.0002	.004	.005	No apparent reaction
Nickel 270, Annealed	Parent	17.02	16.3954	16.3949	.0005	.010	.012	No apparent reaction
Nickel 270, Annealed	Welded	17.02	17.7915	17.7913	.0002	.004	.005	No apparent reaction
Titanium 6Al-4V, STA	Parent	14.72	2.4031	2.4023	.0008	.034	.043	No apparent reaction
Titanium 6Al-4V, STA	Welded	14.72	2.7325	2.7318	.0007	.030	.037	Very slight stain
Titanium 5Al-2.5 Sn, ELI	Parent	14.68	2.3808	2.3804	.0004	.017	.021	No apparent reaction
Titanium 5Al-2.5 Sn, ELI	Welded	14.68	2.5322	2.5313	.0009	.038	.048	No apparent reaction
C-1010 Steel	Parent	14.05	1.3453	1.3448	.0005	.013	.016	Slight blue stain
C-1010 Steel	Welded	14.05	1.9050	1.9043	.0007	.018	.022	Slight darkening
Copper, OFHC	Parent	14.06	1.5519	1.5518	.0001	.002	.003	Slight tarnish
Copper, OFHC	Welded	14.06	3.4312	3.4307	.0005	.011	.014	Slight stain

TABLE 2.1-5

DATA INDICATIVE OF THE COMPATIBILITY OF NITROGEN TRIFLUORIDE WITH VARIOUS METALS

EXPOSURE CONDITIONS: NF_3 Vapor, 344 K, 3.45 MN/m^2 (500 psia) 33 Days
SPECIMEN PRETREATMENTS: Cleaned and Pickled, NF_3 Passivation

Material	Specimen Surface Area, Cm^2	Specimen Weights, g			Penetration Rate		Observations
		Initial	Final	Loss	pm/sec	mpy	
304L SS, Annealed	13.93	0.8388	0.8388	--	0	0	Slight stain
316 ELC SS,	14.21	2.0374	2.0375	(.0001)	0	0	Slight stain
321 SS, Annealed	14.23	2.0732	2.0729	.0003	.009	.012	Considerable stain
347 SS, Annealed	14.06	1.3986	1.3984	.0002	.006	.008	Slight stain
17-4 PH, H-1025	15.66	8.4675	8.4414	.0261	.73	.91	Covered with a black deposit
Inconel 625, Annealed	14.57	3.9567	3.9567	--	0	0	No apparent reaction
Inconel 718, STA	14.06	1.4554	1.4554	--	0	0	Some stain
Monel 400, Annealed	14.21	2.2425	2.2425	--	0	0	Slight stain
Nickel 200, Annealed	14.05	1.5325	1.5324	.0001	.003	.003	Some stain
Nickel 270, Annealed	17.02	16.3288	16.3273	.0015	.035	.043	Some stain
Titanium 6Al-4V, STA	14.72	2.3977	2.3946	.0031	.16	.20	Yellow to gray deposit
Titanium 5Al-2.5 Sn ELI	14.68	2.3782	2.3755	.0027	.14	.18	Yellow to brown deposit
C-1010 Steel	14.05	1.3069	1.3065	.0004	.013	.016	Stained severely
Copper, OFHC	14.06	1.5500	1.5499	.0001	.003	.003	Some tarnish

TABLE 2.1-6

DATA INDICATIVE OF THE COMPATIBILITY OF NITROGEN TRIFLUORIDE WITH VARIOUS METALS

EXPOSURE CONDITIONS: Liquid/Vapor, 233 K, 26 days; 195 K, 15 days
SPECIMEN PRETREATMENTS: Cleaned, No Passivation

Material	Specimen Surface Area, Cm^2	Specimen Weights, g			Penetration rate		Observations
		Initial	Final	Loss	pm/sec	mpy	
304 L SS, Annealed	13.93	0.8426	0.8424	.0002	.005	.006	Some stain
316 ELC SS,	14.21	2.0749	2.0748	.0001	.002	.003	Some stain
321 SS, Annealed	14.23	2.0219	2.0217	.0002	.005	.006	Some stain
347 SS, Annealed	14.06	1.4103	1.4100	.0003	.008	.009	Slight stain
17-4 PH, H-1025	15.66	8.4685	8.4682	.0003	.007	.008	Some stain
Inconel 625, Annealed	14.57	3.9402	3.9400	.0002	.005	.006	Very slight stain
Inconel 718, STA	14.06	1.4438	1.4433	.0005	.012	.015	Considerable stain, greatest in vapor phase
Monel 400, Annealed	14.21	2.2996	2.2993	.0003	.007	.008	Some stain
Nickel 200, Annealed	14.05	1.5611	1.5609	.0002	.005	.006	Some stain, heavier in liquid phase
Nickel 270, Annealed	17.02	16.2041	16.2038	.0003	.006	.007	No apparent reaction
Titanium 6Al-4V, STA	14.72	2.4807	2.4744	.0063	.27	.34	Yellow deposit, heavier in liquid phase
Titanium 5Al-2.5 Sn ELI	14.68	2.3686	2.3620	.0066	.28	.35	Yellow to gray deposit, heavier in liquid phase
C-1010 Steel	14.05	1.3362	1.3358	.0004	.010	.013	Some purple stain
Copper, OFHC	14.06	1.5444	1.5439	.0005	.011	.014	Considerable stain and tarnish

TABLE 2.1-7

DATA INDICATIVE OF THE COMPATIBILITY OF NITROGEN TRIFLUORIDE WITH VARIOUS METALS

EXPOSURE CONDITIONS: Liquid/Vapor, 233 K, 26 days; 195 K, 12 days
SPECIMEN PRETREATMENTS: Cleaned, Pickled, No Passivation

Material	Specimen Surface Area, Cm^2	Specimen Weights, g			Penetration Rates		Observations
		Initial	Final	Loss	pm/sec	mpy	
304 L SS, Annealed	13.93	0.8319	0.8318	--	0	0	No apparent reaction
316 ELC SS,	14.21	2.0565	2.0563	.0002	.005	.007	Very slight stain
321 SS, Annealed	14.23	2.0730	2.0727	.0003	.008	.010	Slight stain
347 SS, Annealed	14.06	1.4114	1.4114	--	0	0	No apparent reaction
17-4 PH, H-1025	15.66	8.3371	8.3371	--	0	0	Very slight stain
Inconel 625, Annealed	14.57	3.9338	3.9338	--	0	0	No apparent reaction
Inconel 718, STA	14.06	1.4730	1.4730	--	0	0	Very slight stain
Monel 400, Annealed	14.21	2.2544	2.2543	.0001	.0002	.0003	Some stain
Nickel 200, Annealed	14.05	1.5186	1.5187	(.0001)	0	0	Some stain
Nickel 270, Annealed	17.02	16.4671	16.4664	.0007	.014	.017	Some stain
Titanium 6Al-4V, STA	14.72	2.3990	2.3932	.0058	.27	.33	Yellow deposit, heavier in liquid phase
Titanium 5Al-2.5 Sn ELI	14.68	2.3682	2.3650	.0032	.15	.18	Brown deposit, heavier in liquid phase
C-1010 Steel	14.05	1.3185	1.3178	.0007	.019	.024	Purple to black coloration
Copper, OFHC	14.06	1.5669	1.5665	.0004	.010	.012	Slightly tarnished

TABLE 2.1-8

DATA INDICATIVE OF THE COMPATIBILITY OF NITROGEN TRIFLUORIDE WITH VARIOUS METALS

EXPOSURE CONDITIONS: Liquid/Vapor, 233 K, 26 days; 195 K, 11 days
SPECIMEN PRETREATMENTS: Cleaned, NF_3 Passivated

Material	Specimen Surface Area, Cm^2	Specimen Weights, g			Penetration Rates		Observations
		Initial	Final	Loss	pm/sec	mpy	
304 L SS, Annealed	13.93	0.8348	0.8348	--	0	0	Very slight stain
316 ELC SS,	14.21	2.0716	2.0713	.0003	.008	.010	Slight stain
321 SS, Annealed	14.23	2.0356	2.0354	.0002	.006	.007	Some staining
347 SS, Annealed	14.06	1.4112	1.4110	.0002	.006	.007	No apparent reaction
17-4 PH, H-1025	15.66	8.4668	8.4666	.0002	.005	.006	No apparent reaction
Inconel 625, Annealed	14.57	3.9377	3.9375	.0002	.005	.006	No apparent reaction
Inconel 718, STA	14.06	1.4642	1.4637	.0005	.013	.017	Considerable stain
Monel 400, Annealed	14.21	2.3078	2.3076	.0002	.005	.006	Considerable stain
Nickel 200, Annealed	14.05	1.5672	1.5670	.0002	.005	.006	Some stain
Nickel 270, Annealed	17.02	16.4597	16.4594	.0003	.006	.008	No apparent reaction
Titanium 6Al-4V, STA	14.72	2.4893	2.4818	.0075	.36	.44	Yellow deposit, heavier in liquid phase
Titanium 5Al-2.5 Sn ELI	14.68	2.3631	2.3509	.0122	.58	.72	Yellow to gray deposit, heavier in liquid phase
C-1010 Steel	14.05	1.3534	1.3532	.0002	.006	.007	Purple stain
Copper, OFHC	14.06	1.5665	1.5658	.0007	.017	.022	Very slight tarnish

2.1, Cleaning and Passivation Pretreatment of Materials (cont.)

The effectiveness of the "passivation treatment" can be assessed by comparison of the data in Tables 2.1-3, 2.1-7, and 2.1-9. Only parent material specimens were used in the tests reported in Tables 2.1-7 and 2.1-9. From the data it is apparent that the rigorous fluorine passivation (Table 2.1-9) does not significantly alter the corrosion resistance compared to the nitrogen trifluoride passivation (Table 2.1-3), and neither one of the "passivation treatments" exhibits a significant improvement in corrosion resistance over the "non-passivated" specimen tabulated in Table 2.1-7. It must be kept in mind at this point that the presence of a "passivation-film" as a result of pre-exposure was not experimentally determined.

In order to determine if the presence of many different materials within a sample bomb can lead to metal/metal interactions which will bias the inherent compatibility of a material with NF_3, a series of tests was conducted with aluminum alloys in contact with and electrically isolated from 304 stainless steel specimens under the exposure conditions already described in the previous test series. The data from the tests is presented in Table 2.1-10.

The data indicate that there is no significant difference in the weight losses that are measured for the aluminum specimens in contact with the 304 stainless steel and the aluminum specimens that are isolated from the 304 stainless steel. The electrical isolation was achieved by placing the ends of the coupons in slots between two Teflon plugs.

Finally a test series was conducted in which only 304 stainless specimens were exposed to nitrogen trifluoride under various conditions in 304-L test bombs. The data are presented in Table 2.1-11. The weight loss values do not differ significantly from the values obtained with 304L specimens used in the multicoupon exposure tests. The welded samples generally exhibit a greater weight loss than the parent samples, but no significant corrosion occurs.

A statistical analysis of the weight changes which all parent metal specimens exhibited after exposure to the liquid/vapor phase nitrogen trifluoride at 233 K was conducted as a function of the pretreatment given prior to exposure. The titanium alloys were analyzed separately because of the large weight losses they incurred in comparison with all the other metal specimens. The results are presented in Table 2.1-12.

TABLE 2.1-9

DATA INDICATIVE OF THE COMPATIBILITY OF NITROGEN TRIFLUORIDE WITH VARIOUS METALS

EXPOSURE CONDITIONS: NF_3 Liquid/Vapor, 233 K, 26 days; 195 K, 15 days
SPECIMEN PRETREATMENTS: Cleaned, Pickled, F_2 Passivation

Material	Specimen Surface Area, Cm^2	Specimen Weights, g			Penetration Rates		Observations
		Initial	Final	Loss	pm/sec	mpy	
304 L SS, Annealed	13.93	0.8448	0.8447	.0001	.002	.003	No apparent reaction
316 ELC SS,	14.21	2.0498	2.0497	.0001	.002	.003	No apparent reaction
321 SS, Annealed	14.23	2.0482	2.0480	.0002	.005	.006	Some stain
347 SS, Annealed	14.06	1.4031	1.4030	.0001	.002	.003	No apparent reaction
17-4 PH, H-1025	15.66	8.4748	8.4747	.0001	.002	.003	Very slight stain
Inconel 625, Annealed	14.57	3.9297	3.9298	(.0001)	0	0	No apparent reaction
Inconel 718, STA	14.06	1.4376	1.4376	--	0	0	Slight stain
Monel 400, Annealed	14.21	2.2575	2.2574	.0001	.002	.003	Some stain
Nickel 200, Annealed	14.05	1.5377	1.5376	.0001	.002	.003	Some stain
Nickel 270, Annealed	17.02	16.2478	16.2478	--	0	0	Some stain
Titanium 6Al-4V, STA	14.72	2.4173	2.4131	.0042	.18	.22	Yellow deposit, heavier in the liquid phase
Titanium 5Al-2.5 Sn ELI	14.68	2.3773	2.3735	.0038	.16	.20	Yellow to gray deposit, heavier in liquid phase
C-1010 Steel	14.05	1.3244	1.3240	.0004	.010	.013	Dark gray coloration
Copper, OFHC	14.06	1.5534	1.5531	.0003	.007	.008	Slightly tarnished

TABLE 2.1-10

DATA INDICATIVE OF THE COMPATIBILITY OF NITROGEN TRIFLUORIDE WITH ALUMINUM ALLOYS IN CONTACT WITH AND ISOLATED FROM 304 STAINLESS STEEL AT VARIOUS CONDITIONS

Specimen Material	Type of Specimen	Specimen Pretreatment	Exposure Condition		Exposure Period*, Days	Surface Area Cm2	Weights, g Initial	Final	Loss	Penetration Rates pm/sec	mpy	Observations
2219 Al T-87	Parent	Cleaned	NF_3 l/v, 223 K	Contact	26 + (18)	15.13	1.6691	1.6691	--	0	0	Slight stain in liquid phase
2219 Al T-87	Parent	Pickled		Isolated		15.13	1.7112	1.7112	--	0	0	Very slight stain in liquid phase
6061 Al T-6	Parent	NF_3 Passivation		Contact		14.42	0.4804	0.4805	(.0001)	0	0	No apparent reaction
6061 Al T-6	Parent			Isolated		14.42	0.4855	0.4856	(.0001)	0	0	Very slight stain in liquid phase
304 SS	Parent					14.05	1.3825	1.3825	--	0	0	No apparent reaction
2219 Al T-87	Welded	Cleaned	NF_3 l/v, 223 K	Contact	26 + (17)	15.13	1.7735	1.7734	.0001	.007	.008	Some stain in liquid phase
2219 Al T-87	Welded	Pickled		Isolated		15.13	1.7222	1.7219	.0003	.020	.025	No apparent reaction
304 SS	Parent	NF_3 Passivation				14.05	1.3732	1.3731	.0001	.007	.008	Very slight stain in liquid phase
6061 Al T-6	Welded	Cleaned	NF_3 l/v, 223 K	Contact	26 + (19)	14.42	0.8381	0.8378	.0003	.020	.025	No apparent reaction
6061 Al T-6	Welded	Pickled		Isolated		14.42	0.8664	0.8663	.0001	.007	.008	No apparent reaction
304 SS	Parent	NF_3 Passivation				14.05	2.0890	2.0888	.0002	.005	.006	No apparent reaction
6061 Al T-6	Parent	Cleaned	NF_3 v, 223 K	Contact	26 + (20)	14.42	0.4906	0.4905	.0001	.007	.008	No apparent reaction
6061 Al T-6	Parent	Pickled		Isolated		14.42	0.4841	0.4841	--	0	0	No apparent reaction
304 SS	Parent	NF_3 Passivation				14.05	1.3807	1.3807	--	0	0	No apparent reaction
2219 Al T-87	Parent			Contact		15.13	1.7160	1.7158	.0002	.013	.016	Very slight stain in liquid phase
2219 Al T-87				Isolated		15.13	1.7012	1.7012	--	0	0	No apparent reaction
6061 Al T-6	Welded	Cleaned	NF_3 v, 223 K	Contact	26 + (20)	14.42	0.9797	0.9794	.0003	.019	.023	No apparent reaction
6061 Al T-6	Welded	Pickled		Isolated		14.42	0.7684	0.7682	.0002	.013	.016	No apparent reaction
304 SS	Parent	NF_3 Passivation				14.05	1.8752	1.8750	.0002	.005	.006	No apparent reaction
2219 Al T-87	Welded	Cleaned	NF_3 v, 223 K	Contact	26 + (19)	15.13	1.8592	1.8589	.0003	.019	.023	Very slight stain
2219 Al T-87	Welded	Pickled		Isolated		15.13	1.5380	1.5379	.0001	.006	.008	Very slight stain
304 SS	Parent	NF_3 Passivation				14.05	1.3678	1.3677	.0001	.002	.003	Slight stain on contact side
2219 Al T-87	Parent	Cleaned	NF_3 l/v, 223 K	Contact	26 + (19)	15.13	1.7130	1.7127	.0003	.019	.023	No apparent reaction
2219 Al T-87	Parent	Pickled		Isolated		15.13	1.6918	1.6916	.0002	.013	.016	No apparent reaction
6061 Al T-6	Parent	No Passivation		Contact		14.42	0.4897	0.4897	--	0	0	No apparent reaction
6061 Al T-6	Parent			Isolated		14.42	0.4842	0.4841	.0001	.007	.008	No apparent reaction
304 SS	Parent					14.05	1.3815	1.3815	--	0	0	Some tarnish on contact side with 6061 Al
6061 Al T-6	Parent	Cleaned	NF_3 l/v, 223 K	Contact	26 + (16)	14.42	0.4860	0.4859	.0001	.007	.009	No apparent reaction
6061 Al T-6	Parent	Pickled		Isolated		14.42	0.4556	0.4554	.0002	.014	.017	No apparent reaction, except for slight stain
304 SS	Parent	F_2 Passivation				14.05	1.3916	1.3917	--	0	0	No apparent reaction
6061 Al T-6	Parent	Cleaned	NF_3 l/v, 223 K	Contact	26 + (16)	14.42	0.4850	0.4848	.0002	.014	.017	No apparent reaction
6061 Al T-6	Parent	No Passivation		Isolated		14.42	0.4910	0.4908	.0002	.014	.017	No apparent reaction
304 SS	Parent					14.05	1.3621	1.3620	.0001	.002	.003	Some stain
2219 Al T-87	Parent	Cleaned	NF l/v, 223 K	Contact	26 + (19)	15.13	1.7016	1.7014	.0002	.013	.016	Very slight stain on contact side
2219 Al T-87	Parent	NF_3 Passivation		Isolated		15.13	1.6780	1.6778	.0002	.013	.016	No apparent reaction
304 SS	Parent					14.05	1.3832	1.3832	--	0	0	Slight stain on contact sides with 6061 and 2219
6061 Al T-6	Parent			Contact		14.42	0.4826	0.4824	.0002	.013	.016	No apparent reaction
6061 Al T-6	Parent			Isolated		14.42	0.4823	0.4820	.0003	.019	.024	No apparent reaction
6061 Al T-6	Parent	Cleaned	NF_3 v, 500 psig, 344 K	Contact	33	14.42	0.4811	0.4810	.0001	.009	.011	Some stain
6061 Al T-6	Parent	Pickled		Isolated		14.42	0.4865	0.4864	.0001	.009	.011	Some stain
304 SS	Parent	NF_3 Passivation				14.05	1.3883	1.3882	.0001	.003	.004	Some stain and tarnish on contact side

*Values in parentheses are the number of days in which the samples were stored at 195K.

TABLE 2.1-11

DATA INDICATIVE OF THE COMPATIBILITY OF NITROGEN TRIFLUORIDE WITH 304 STAINLESS STEEL AT VARIOUS CONDITIONS

Type of Specimen	Specimen Pretreatment	Exposure Condition	Exposure Period,* days	Surface Area Cm2	Weights, g Initial	Weights, g Final	Weights, g Loss	Penetration Rates pm/sec	Penetration Rates mpy	Observations
Parent	Cleaned, Pickled, NF_3 Passivation	-50°C, 1/v	26, (19)	14.06	1.3873	1.3873	--	0	0	Some stain
Welded			26, (19)	14.06	2.0595	2.0590	.0005	.011	.014	Some stain
Parent	Cleaned, Pickled, NF_3 Passivation	-50°C, V_a	26, (12)	14.06	1.3753	1.3570	.0003	.008	.010	Very slight stain
Welded			26, (12)	14.06	1.7675	1.7670	.0005	.013	.017	Very slight stain
Parent	Cleaned, Pickled, No Passivation	-50°C, 1/v	26, (12)	14.06	1.3896	1.3896	--	0	0	No apparent reaction
Welded			26, (12)	14.06	1.9266	1.9264	.0002	.005	.007	No apparent reaction
Parent	Cleaned, Pickled, F_2 Passivation	-50°C, 1/v	26, (16)	14.06	1.3808	1.3807	.0001	.002	.003	No apparent reaction
Welded			26, (16)	14.06	1.9430	1.9426	.0004	.010	.012	No apparent reaction
Parent	Cleaned, No Passivation	-50°C, 1/v	26, (16)	14.06	1.3820	1.3819	.0001	.002	.003	No apparent reaction
Welded			26, (16)	14.06	1.9669	1.9661	.0008	.020	.024	No apparent reaction
Parent	Cleaned, NF_3 Passivation	-50°C, 1/v	26, (19)	14.06	1.3626	1.3626	--	0	0	Slightly stained
Welded			26, (19)	14.06	2.1699	2.1699	--	0	0	No apparent attack
Parent	Cleaned, Pickled, NF_3 Passivation	71°C, v	33	14.06	1.3628	1.3626	.0002	.006	.008	Some tarnish
Welded			33	14.06	1.9550	1.9547	.0003	.009	.012	Some tarnish
Parent	Cleaned, Pickled, NF_3 Passivation	71°C, NF_3 1/v H_2O	33	14.06	1.3783	1.3572	.0211	.66	.81	Major deposits in vapor phase, greater attack in liquid phase
Welded			33	14.06	1.7592	1.7319	.0273	.85	1.05	Major deposits in vapor phase
Parent	Cleaned, Pickled, No Passivation	71°C, 1/v H_2O	33	14.06	1.3768	1.3768	--	0	0	No apparent attack
Welded	Cleaned, Pickled, No Passivation		33	14.06	2.0704	2.0703	.0001	.003	.004	No apparent attack

*Values in parentheses are the number of days in which the samples were stored at -78°C.

TABLE 2.1-12

STATISTICAL ANALYSIS OF WEIGHT CHANGES OF PARENT METAL SPECIMENS SUBJECTED TO VARIOUS PRETREATMENTS PRIOR TO EXPOSURE TO LIQUID/VAPOR NITROGEN TRIFLUORIDE AT 223 K

	Mean Weight Change, mg	
Pretreatment Procedure	Titanium Alloys	All Other Metals
Cleaned only	6.4 ± .2	0.26 ± .13
Cleaned, Pickled, No Preexposure	4.5 ± 1.8	0.094 ± .14
Cleaned, Preexposed to NF_3	9.8 ± 3.3	0.23 ± .17
Cleaned, Pickled, Preexposed to NF_3	3.0 ± 0.6	0.056 ± .12
Cleaned, Pickled, Preexposed to F_2	4.0 ± 0.3	0.11 ± .12

The data in Table 2.1-12 indicate that pickling after cleaning does reduce the weight changes which occur during exposure to nitrogen trifluoride, but that pre-exposure to nitrogen trifluoride or fluorine at ambient temperatures after pickling does not significantly enhance the metal/nitrogen trifluoride compatibility. Based on this data, the metal specimens subjected to long-term exposure to nitrogen trifluoride were cleaned and pickled, but were not pre-exposed to nitrogen trifluoride as a passivation step.

Chemical analyses of the nitrogen trifluoride recovered from the tests reported in Tables 2.1-3 through 2.1-11 were conducted using the method described in "Analysis of Nitrogen Trifluoride", AFRPL-TR-76-89, December 1976 using gas chromatography and a specific ion electrode to determine the total active fluorides.

The test bombs which contained "all metals except Al" were fitted with valves which allowed the nitrogen trifluoride to be sampled directly. All the other test bombs were opened in a helium atmosphere with the liquid nitrogen trifluoride condensed in a liquid nitrogen bath and the contents were transferred to an appropriate size sample bomb for the analysis. The analyses obtained with direct sampling are less subject to sample handling contamination and thus are more reliable. In some cases samples were lost prior to analysis. Because nitrogen trifluoride was vapor-transferred from the original sample containers, an attempt was made to recover the solid materials which remained by washing the opened sample containers with Freon and weighing the residues after the Freon had evaporated. In some cases no data was obtained and it is so specified in the tabulation. The data are presented in Table 2.1-13.

TABLE 2.1-13

ANALYSES OF NITROGEN TRIFLUORIDE EXPOSED TO VARIOUS TEST CONDITIONS AND MATERIALS

Type of Exposure	Composition, Weight Percent: NF_3	Active Fluorides as HF	N_2	CO/O_2	CF_4	CO_2	N_2O	Total Solids Recovered	Table No. in Which Specimen Data are Reported	Metal Pretreatment
NF_3 as received, cyl 17341-C in September	98.3	.077	0.31	0.74	0.38	.03	.013			
Cylinder 17341-C, December analysis	98.2	.035	0.59	0.17	0.58	.05	0.42			
Liquid/vapor NF_3 223 K, 26 days, 195 K - 20 days, all metals except Al	98.1	.031	0.25	0.71	0.75	.04	0.13	0.5 mg	2.1-3	Cleaned, Pickled, NF_3 Passivated
500 psig vapor NF_3, 223 K - 26 days, 195 K - 15 days, all metals except Al	98.7	.015	0.14	0.33	0.67	.05	0.11	No Data	2.1-4	Cleaned, Pickled, NF_3 Passivated
Liquid/vapor NF_3, 223 K - 26 days, 195 K - 18 days, aluminum and 304	98.3	.018	0.22	0.61	0.68	.05	0.14	0.6 mg	2.1-10	Cleaned, Pickled, NF_3 Passivated
Liquid/vapor NF_3, 223 K - 26 days, 195 K - 19 days, aluminum and 304	98.2	.018	0.25	0.68	0.64	.05	0.13	0.3 mg	2.1-10	Cleaned, Pickled, NF_3 Passivated
Liquid/vapor NF_3, 223 K - 26 days, 195 K - 17 days, aluminum and 304	98.2	.021	0.25	0.70	0.68	.05	0.11	None	2.1-10	Cleaned, Pickled, NF_3 Passivated
Liquid/vapor NF_3, 223 K - 26 days, 195 K - 12 days, all metals except Al	98.1	.053	0.27	0.65	0.73	.05	0.12	No Data	2.1-7	Cleaned, Pickled
Liquid/vapor NF_3, 223 K - 26 days, 195 K - 19 days, aluminum and 304	98.3	.031	0.19	0.64	0.65	.03	0.15	None	2.1-10	Cleaned, Pickled
Liquid/vapor NF_3, 223 K - 26 days, 195 K - 12 days, 304 only	98.0	.042	0.27	0.79	0.71	.04	0.11	No Data	2.1-11	Cleaned, Pickled
Liquid/vapor NF_3, 223 K - 26 days, 195 K - 15 days, all metals except Al	98.2	.043	0.26	0.63	0.71	.04	0.13	No Data	2.1-9	Cleaned, Pickled, F_2 Passivated
Liquid/vapor NF_3, 223 K - 26 days, 195 K - 16 days, 304 only	98.2	.040	0.31	0.63	0.72	.04	0.11	None	2.1-11	Cleaned, Pickled, F_2 Passivated
Liquid/vapor NF_3, 223 K - 26 days, 195 K - 15 days, all metals except Al	98.1	.033	0.25	0.68	0.71	.05	0.14	0.9 mg	2.1-6	Cleaned
Liquid/vapor NF_3, 223 K - 26 days, 195 K - 16 days, aluminum and 304	98.5	.020	0.10	0.51	0.69	.05	0.12	0.1 mg	2.1-10	Cleaned
Liquid/vapor NF_3, 223 K - 26 days, 195 K - 16 days, 304 only	98.[illegible]	.033	0.31	0.66	0.66	.04	0.11	0.4 mg	2.1-11	Cleaned
Liquid/vapor NF_3, 223 K - 26 days, 195 K - 11 days, all metals except Al	98.2	.038	0.26	0.63	0.74	.04	0.14	0.5 mg	2.1-8	Cleaned, NF_3 Passivated
Liquid/vapor NF_3, 223 K - 26 days, 195 K - 19 days, aluminum and 304	98.0	.016	0.33	0.87	0.66	.05	0.12	0.3 mg	2.1-10	Cleaned, NF_3 Passivated
Liquid/vapor NF_3, 223 K - 26 days, 195 K - 9 days, 304 only	98.2	.027	0.22	0.73	0.73	.03	0.10	None	2.1-11	Cleaned, NF_3 Passivated
500 psia NF_3 vapor, 344 K - 33 days all metals except Al	97.2	.393	0.66	0.92	0.62	.06	0.17	No Data	2.1-5	Cleaned, Pickled, NF_3 Passivated
500 psia NF_3 vapor, 344 K - 33 days, aluminum and 304	97.8	.266	0.47	0.69	0.61	.05	0.11	No Data	2.1-10	Cleaned, Pickled, NF_3 Passivated
500 psia NF_3 vapor, 344 K - 33 days, 304 only	98.2	.019	0.35	0.66	0.60	.07	0.11	No Data	2.1-11	Cleaned, Pickled, NF_3 Passivated

The significant items to note from the data are: (1) the nitrogen trifluoride used in the 223 K static storage tests is virtually unchanged, (2) at 344 K the nitrogen trifluoride stored with "all metals except Al" exhibited a decrease of 1% in nitrogen trifluoride and a significant increase in active fluoride content (the titanium samples and the 17-4 PH sample exhibited significant weight losses); the "aluminum and 304" sample exhibits a smaller decrease in nitrogen trifluoride content and increase in active fluoride while the "304 only" sample shows no change. Based on the data, the titanium and 17-4 PH specimens were exposed to nitrogen trifluoride vapor at elevated temperatures for prolonged periods in isolated test bombs.

The results can be summarized as follows.

1. No metal specimen exposed to either liquid or vapor nitrogen trifluoride at 223 K (-50°C) for a month, or to nitrogen trifluoride vapor at 3.45 MN/m^2 (500 psia) at 344 K (160 F) for a month exhibited a corrosion penetration rate of greater than 0.8 pm/sec (1 mil per year).

2. The pickling of the metal samples provided a very slight improvement in the corrosion resistance of the materials.

3. Pre-exposure of the metal specimens to nitrogen trifluoride or fluorine at room temperature did not alter the corrosive behavior as compared to no pre-exposure of the metal specimens.

4. No intermetallic interactions or galvanic corrosion occurred in uncontaminated liquid NF_3.

5. No significant compositional changes in nitrogen trifluoride occurred at the lower temperatures and only minimal compositional changes were detected at 344 K.

2.1.2 Cleaning and Passivation Pretreatment of Non-Metallic Materials

The purpose of this task was to establish early in the experimental program the appropriate procedures for preparation of non-metallic material surfaces prior to exposure to nitrogen trifluoride for prolonged periods of time.

2.1, Cleaning and Passivation Pretreatment of Materials (cont.)

2.1.2.1 Cleaning Procedures for Non-Metallic Materials

The non-metal specimens were, except for the carbon, the greases, and the epoxy, detergent washed with a Turco Playdit solution, rinsed with deionized water and vacuum dried overnight at 333 K (140 F). The carbons, the greases, and the epoxy were used in the as-received condition.

2.1.2.2 Passivation Procedure for Non-Metallic Materials

Although passivation per se of non-metals is inappropriate, the exposure of non-metals after cleaning and drying to nitrogen trifluoride at ambient temperature and pressure may have merit in regard to removal or reaction with absorbed or occluded species. We had planned to evaluate this procedure by exposing non-metal samples for the initial non-metal screening tests to nitrogen trifluoride vapor at room temperature for several hours prior to the testing. The results obtained with nitrogen trifluoride-exposed samples were then to be compared to the results obtained with the samples which were not subjected to the pretreatment. On the basis of these results, the appropriate pretreatment was to be selected. However, the initial screening tests with non-metallic materials with no pre-exposure to nitrogen trifluoride exhibited no gross incompatibility between the nitrogen trifluoride and non-metallic candidates at temperatures up to 478 K (400 F) except for the Epoxy EA-384 and Silastic LS-53 during 5-15 minute exposure periods.

2.1.2.3 Test Apparatus and Procedures

A photograph of the assembled test apparatus is shown in Figure 2.1.4. The test apparatus consists of a reaction flask which contains a copper plate which is heated from beneath by electrical resistance coils. The lower section of the glass flask is filled with nitrogen to prevent the nitrogen trifluoride from reacting with the hot resistance wire. The top half of the glass flask ($\sim$480ml volume) is initially filled with nitrogen which is displaced by the nitrogen trifluoride as its flow is initiated. The surface of the copper plate is shown in Figure 2.1.5. The nitrogen trifluoride is heated as it passes through a 0.32 cm (.125 in.) copper coil which is located immediately beneath the copper plate. The flowing gas impinges on the surface of the non-metal specimen and a thermocouple is placed on the surface of the non-metal specimen to measure the temperature. The output from the thermocouple is recorded on the Y-axis of an X-Y recorder. Another thermocouple is placed on the surface of the copper plate and the output is recorded on the X-axis of the X-Y recorder. In this manner as the sample is heated, any endotherm or exotherm is detected by deviations in slope of the recorded trace.

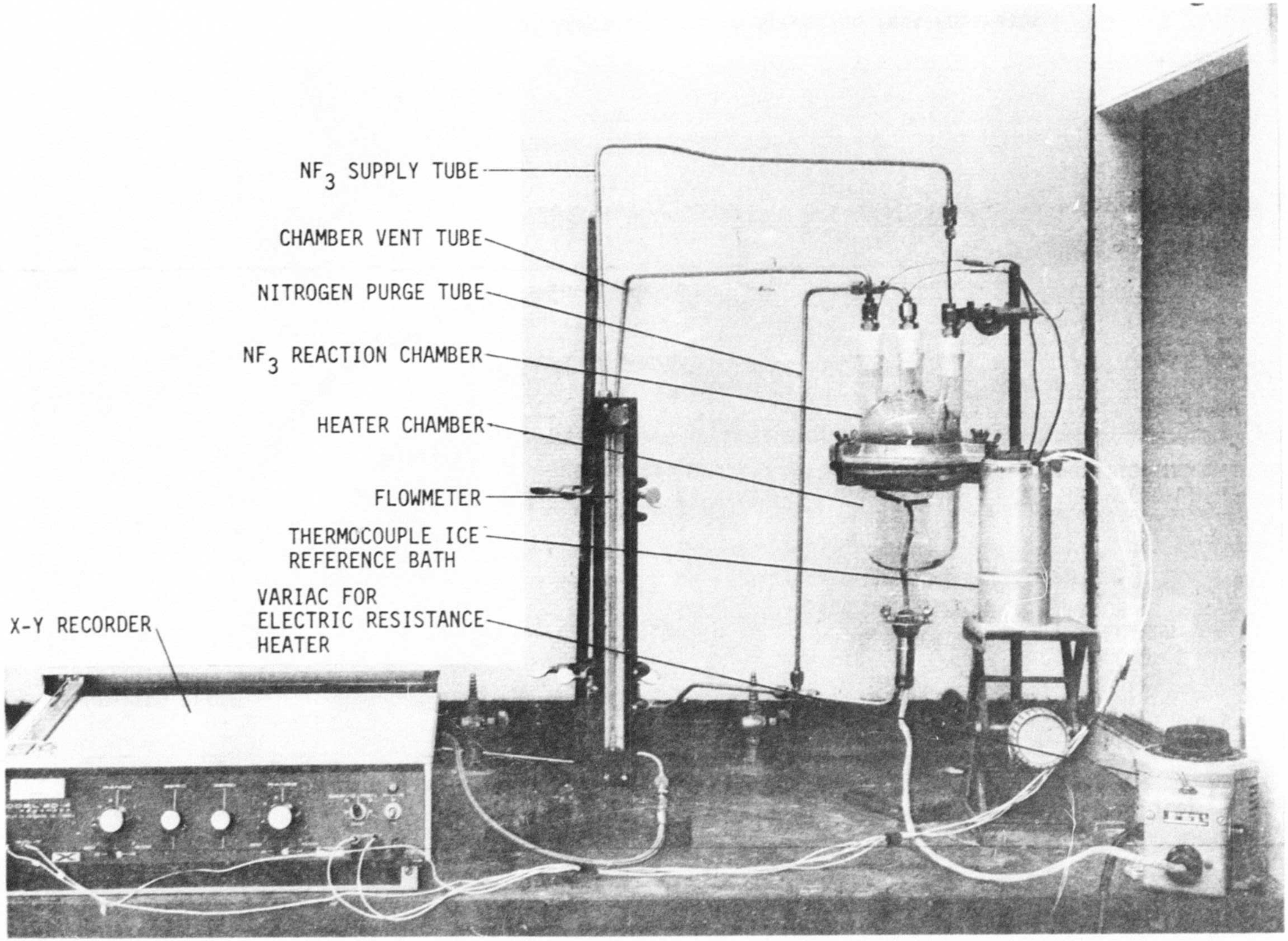


Figure 2.1.4. Apparatus for Non-Metal/Nitrogen Trifluoride Compatibility Screening Tests

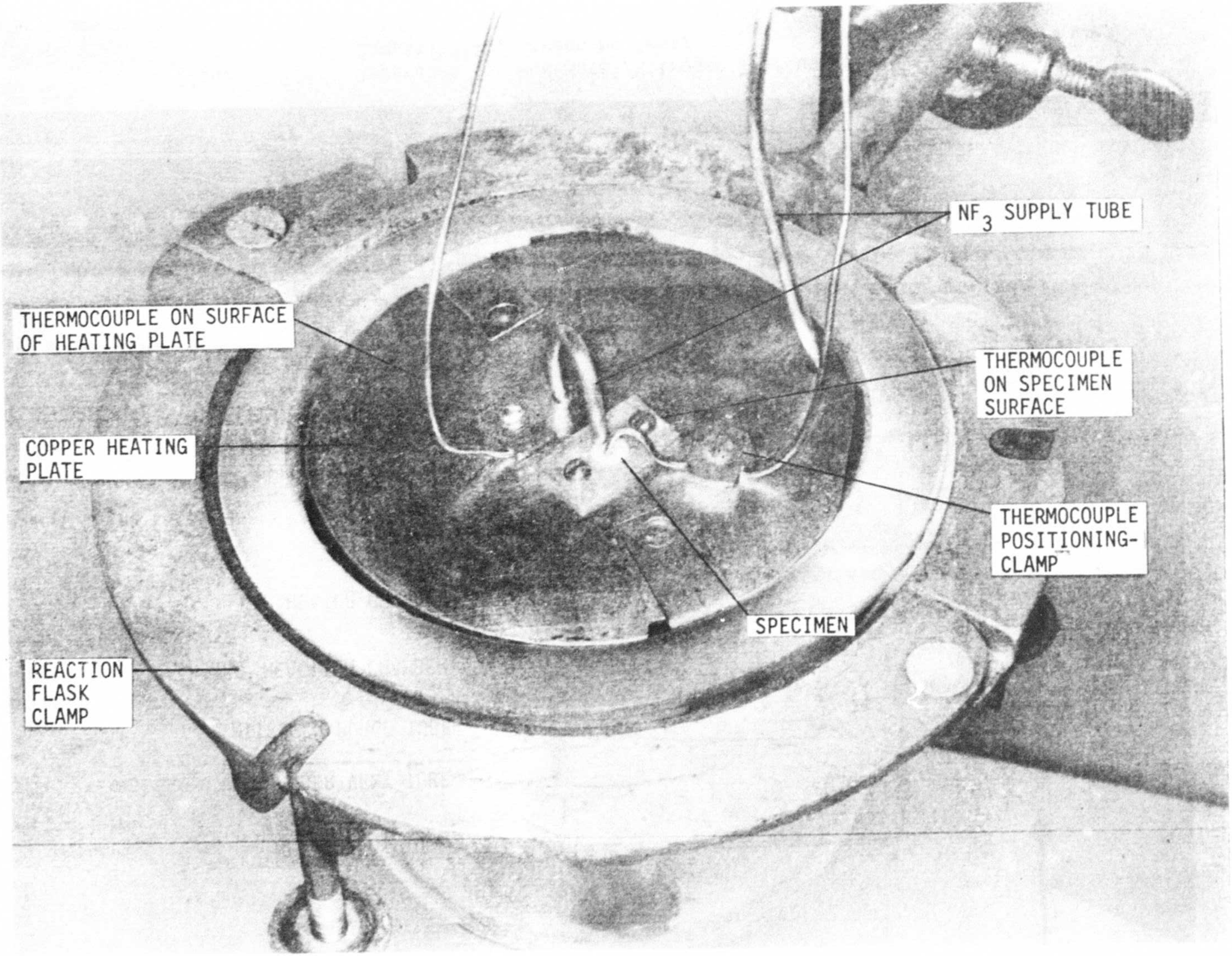


Figure 2.1.5. Photograph of Reaction Zone in Screening Test Apparatus

Two types of tests were conducted with the apparatus. In the first test series the specimens were heated from ambient temperature to 478 K (400°F) in a period of 10 to 15 minutes while either oxygen or nitrogen trifluoride was flowing at a rate of 60 ml/min onto the surface of the specimen. In addition to the thermal measurements, visual changes in the specimens were noted. The total pressure in the reaction flask was one atmosphere. In the second test series, the specimens were heated while nitrogen trifluoride was flowing onto the surface at a rate of 60 ml/min to the maximum usage temperature recommended for the material and the specimen was held at that temperature for a period of five minutes while the nitrogen trifluoride flow rate remained a 60 ml/minute.

2.1.2.4 Test Results

The results of the tests are presented in Table 2.1-14. The tests in which nothing occurred are identified by a minus sign (-); the tests in which something occurred are designated by a plus sign (+) and the phenomena are identified. The temperatures reported in the table are the values for the exposed surface of the specimens. The nitrogen trifluoride used in the tests had a minimum purity level of 98.2 weight percent NF_3 and a maximum active fluoride content of 0.17 weight percent.

The significant items to note from the test results are: (1) no apparent reaction occurred between NF_3 and the non-metals except for Mylar at 478 K (400 F), Epoxy (EA-934) at 450 K (350 F), and Silastic LS-53 at 433 K (320 F); (2) the reactivity of nitrogen trifluoride with the non-metals is comparable to that of oxygen under the same conditions with the exception of the above mentioned materials. The reader should be cautioned that the preceding tests are screening tests for gross incompatibility under the described test condition and are not necessarily an indication of compatibility during long-term exposure.

TABLE 2.1-14

DATA INDICATIVE OF THE REACTIVITY OF NON-METALS WITH NITROGEN TRIFLUORIDE AT ONE ATMOSPHERE PRESSURE AND IN COMPARISON WITH GASEOUS OXYGEN

Material	Ambient to 478K (400F) Test		Maximum Usage Temperature For Five Minutes in NF_3			Phenomena Detected
	O_2	NF_3	Reaction	°K	°F	
Polytetrafluoroethylene	-	-	-	533	500	None
FFP Teflon	-	-	-	478	400	None
PFA Teflon	-	-	-	478	400	None
KEL-F 81 CTFE	-	-	-	478	400	None
Neoprene	-	-	-	450	350	None
Tygon	455K (360F)	456K (361F)	+	366	200	Darkening in Coloration
Lucite	428K (310F)	448K (346F)	-	394	250	Melting
Polyethylene	371K (208F)	371K (208F)	-	366	200	Melting
Polypropylene	402K (264F)	416K (290F)	-	394	250	Melting
Kevlar	-	-	-	533	500	None
Krytox	-	-	-	478	400	None
Dry Powder TFE (MS-122)	-	-	-	478	400	None
AF-E-124 (DuPont ECD-006)	-	-	-	589	600	None
Mylar	-	478K (400F)	-	450	350	Slight Color Change
Epoxy (EA-934)	-	450K (350F)	+	533	500	Coloration Change from Gray to Reddish-Brown
Carbon CJPS	-	-	-	589	600	None
Fluorosilicone (FS 3451)	-	-	-	478	400	None
Oil-Soaked (Nujol Mineral Oil) Kimwipe	-	-				Kimwipe Gradually turned Brown
Viton (MIL-R-83248 Class 1)	-	-	+	533	500	Slight Black Deposit
Silastic LS-53	-	433K (320F)	+	533	500	Slight Endotherm at 433K (320F). At 533K (500F) Slight Color Change and Hardening Occurred.

2.2 STATIC EXPOSURE TESTS

The purpose of the static exposure tests was to determine the degree of chemical compatibility which exists between nitrogen trifluoride and the various candidate materials under static conditions.

2.2.1 Static Exposure Tests with Metals

2.2.1.1 Apparatus and Procedures

The metal specimens used in the tests were of the same dimensions as described in Section 2.1.1.3 and the specimen containers were the same as described in Section 2.1.1.3 and shown in Figures 2.1.1, 2.1.2, and 2.1.3. Based on the results reported in Section 2.1.1.4, prior to testing, the metal specimens were washed in detergent solution (Turco Plaudit), degreased in an isopropanol bath, rinsed with deionized water, then immersed in the appropriate pickling solution listed in Table 2.1-1, rinsed with deionized water, rinsed with isopropanol, and finally dried under vacuum at 333 K (140 F). There was no pre-exposure of the specimens to gaseous nitrogen trifluoride prior to the loading with the desired quantity of nitrogen trifluoride for the test condition.

The following metals were tested in separate 304L stainless steel containers as shown in Figure 2.1.3: (1) the aluminum alloys, (2) the titanium alloys, (3) aluminum bronze 623, (4) tungsten-2% thoria, (5) beryllium copper, (6) CRES 17-4PH, H-1025, (7) C-1010 steel and (8) Carpenter Custom 455. The remainder of the metal alloys were exposed in ganged-fashion as shown in Figure 2.1.1 and 2.1.2. Typical compositions of the metal candidates evaluated are presented in Appendix A.

The static test durations were for periods of 30 days, 90 days, and 270 days. The varying exposure periods allowed the nature of the reaction which might occur to be evaluated and provided some realistic corrosion penetration rate data.

Liquid/vapor exposure tests were conducted at 195 K (-78 C) because the temperature could be reliably maintained using evaporating solid CO_2 as the refrigerant, the vapor pressure of the liquid nitrogen trifluoride is significant 1.4 MN/m^2 (200 psia), and the liquid/metal reaction rates should be greater than in a boiling liquid nitrogen environment 77 K (-196 C).

Gaseous exposure tests were conducted at 344 K (160 F) because the temperature represents the upper limit which is likely to be encountered at earth surface conditions. The gaseous tests were conducted at pressures ranging from 3.45 MN/m^2 (500 psia) for all the materials to 17.24 MN/m^2 (2500 psia) for selected materials.

The static compatibility test matrix for the metals is presented in Table 2.2-1. The numbers in the test matrix table should be interpreted as follows. Number 1 indicates one test specimen of parent material. Number 2 indicates that both a welded and a parent metal specimen are tested.

After the tests were completed, the nitrogen trifluoride was recovered from the test containers and analyzed to determine the compositional changes which may have occurred during the exposure.

2.2.1.2 Experimental Results

The experimental results obtained from the liquid/vapor static exposure tests for durations of 30, 90 and 270 days at 195 K (-78 C) are tabulated in Table 2.2-2. The weight changes, corrosion penetration rate values, initial active fluoride content of the nitrogen trifluoride and visual observations are recorded in the table. The initial active fluoride values are important because one of the active fluoride species, hydrogen fluoride, is shown to significantly increase the corrosion of metals in nitrogen trifluoride (see Section 2.14).

The significant items to note from the data in Table 2.2-2 after 270 days exposure are as follows: (1) none of the metal alloys exhibited any significant corrosion, the maximum corrosion penetration rate observed is less than 0.08 pm/sec (0.1 mpy); (2) the aluminum alloys corrosion penetration rates ranged from 0.006 to 0.063 pm/sec (0.008 to 0.078 mpy); and (3) the remainder of the metals except for copper OFHC (0.04 pm/sec, 0.05 mpy) and Inconel 718 (0.014 pm/sec, 0.018 mpy) exhibited rates of less than 0.008 pm/sec (0.010 mpy). On a comparison basis, the 270 day-exposure corrosion data for the aluminum alloys generally are higher values than were obtained for the 30 and 90 day exposures. The nitrogen trifluoride which was used for the 270 day exposure tests contained 0.10 weight percent active fluoride calculated as HF while the nitrogen trifluoride used for the 30 and 90 day tests contained less than 0.0001 weight percent active fluoride. This difference in active fluoride content can produce the difference in corrosion rates which are reported.

The experimental results obtained from the gaseous static exposure tests for durations of 30, 90 and 270 days at 344 K (160 F) and 3.45 MN/m^2 (500 psia) are presented in Table 2.2-3.

The significant items to note from the data obtained from the metal specimens exposed to nitrogen trifluoride for 270 days at 344 K (160 F) and 3.45 MN/m^2 (500 psia) are as follows: (1) none of the metal alloys except for Carpenter Custom 455 (0.34 pm/sec, 0.43 mpy),

TABLE 2.2-1

STATIC COMPATIBILITY TEST MATRIX FOR METALS

Material	Vapor/Liquid NF_3 Exposure -78°C, ~200 psia			Gaseous NF_3 Exposures 160°F, 500 psia			160°F, 1500 psia			160°F, 2500 psia		
	30 Days	90 Days	270 Days	30 Days	90 Days	270 Days	30 Days	90 Days	270 Days	30 Days	90 Days	270 Days
Al 2219, T-37	2	2	2	2	2	2	2	2	2	2	2	2
Al 6061, T-6	2	2	2	2	2	2						
Al 2014, T-6	2	2	2	2	2	2						
Al 1100	2	2	2	2	2	2						
301 SS				2	2	2	2	2	2	2	2	2
304 SS, Annealed	2	2	2	2	2	2						
304L SS, Annealed	2	2	2	2	2	2						
316 ELC SS, Annealed	2	2	2	2	2	2						
321 SS, Annealed	2	2	2	2	2	2						
347 SS, Annealed	2	2	2	2	2	2						
17-4PH, H-1025	2	2	2	2	2	2						
Inconel 625, Annealed	2	2	2	2	2	2						
Inconel 718, STA	2	2	2	2	2	2	2	2	2	2	2	2
Monel 400, Annealed	2	2	2	2	2	2						
Nickel 200, Annealed	2	2	2	2	2	2						
Nickel 270, Annealed	2	2	2	2	2	2						
Ti 6Al-4V, STA	2	2	2	2	2	2	2	2	2	2	2	2
Ti 5Al-2.5 Sn	2	2	2	2	2	2						
1020 Steel	-	-	-	2	2	2	2	2	2	2	2	2
Cu OFHC, Annealed	2	2	2	2	2	2						
Nitronic 40	2	2	2	2	2	2						
Maraging Steel 200	2	2	2	2	2	2	2	2	2	2	2	2
Maraging Steel 250	2	2	2	2	2	2	2	2	2	2	2	2
Carpenter Custom 455	2	2	2	2	2	2						
Aluminum Bronze 623	1	1	1	1	1	1						
A-286 SS	1	1	1	1	1	1						
Tungsten	1	1	1	1	1	1						
303 SS	1	1	1	1	1	1						
Beryllium Copper	1	1	1	1	1	1						

TABLE 2.2-2

DATA INDICATIVE OF THE COMPATIBILITY OF LIQUID/VAPOR PHASE NITROGEN TRIFLUORIDE AT 195 K (-78 C) WITH VARIOUS METALS

Material	Specimen Type	Exposure Time, Days	Specimen Surface Area, cm²	Specimen Weights, gm Initial	Final	Loss	Penetration Rates pm/sec	mpy	Test No.	Initial Active Fluoride W/O	Observations
1100 Aluminum	Parent	33	14.38	0.4909	0.4908	0.0001	0.009	0.011	A1X	.0001	No apparent reaction
	Welded	33	14.35	0.7100	0.7100	0.0000	0	0	A1X	.0001	No apparent reaction
	Parent	90	14.22	0.4905	0.4905	0	0	0	A1Y	.0001	No apparent reaction
	Welded	90	14.23	0.7343	0.7343	0	0	0	A1Y	.0001	No apparent reaction
	Parent	271	14.13	0.4873	0.4849	0.0024	0.027	0.033	A1Z	.10	Slight tarnish
	Welded	271	14.41	0.7008	0.6991	0.0017	0.019	0.023	A1Z	.10	Slight tarnish
2014, T6 Aluminum	Parent	33	16.42	4.0781	4.0781	0	0	0	A1X	.0001	Very, very slight stain
	Welded	33	16.48	4.4346	4.4346	0	0	0	A1X	.0001	Very, very slight stain
	Parent	90	16.51	4.1110	4.1109	0.0001	0.003	0.004	A1Y	.0001	No apparent reaction
	Welded	90	16.54	4.3839	4.3837	0.0002	0.006	0.007	A1Y	.0001	No apparent reaction
	Parent	271	16.42	4.0909	4.0897	0.0012	0.011	0.014	A1Z	.10	Very slight tarnish
	Welded	271	16.48	4.3226	4.3194	0.0032	0.031	0.038	A1Z	.10	Very slight tarnish
2219, T87 Aluminum	Parent	33	15.35	1.6436	1.6436	0	0	0	A1X	.0001	No apparent reaction
	Welded	33	15.00	1.9041	1.9040	0.0001	0.009	0.011	A1X	.0001	No apparent reaction
	Parent	90	14.83	3.4425	3.4423	0.0002	0.006	0.008	A1Y	.0001	No apparent reaction
	Welded	90	14.63	3.6917	3.6911	0.0006	0.020	0.024	A1Y	.0001	No apparent reaction
	Parent	271	15.39	1.6591	1.6585	0.0006	0.006	0.008	A1Z	.10	Slight tarnish
	Welded	271	15.15	1.9378	1.9348	0.0030	0.031	0.039	A1Z	.10	Slight tarnish
6061 T6 Aluminum	Parent	33	14.51	0.4763	0.4763	0	0	0	A1X	.0001	No apparent reaction
	Welded	33	14.71	0.7610	0.7610	0	0	0	A1X	.0001	No apparent reaction
	Parent	90	14.39	0.4800	0.4798	0.0002	0.007	0.008	A1Y	.0001	Some tarnish
	Welded	90	14.57	0.7352	0.7351	0.0001	0.003	0.004	A1Y	.0001	Some tarnish
	Parent	271	14.42	0.4681	0.4666	0.0015	0.016	0.020	A1Z	.10	Slight tarnish with a clear streak (both phases)
	Welded	271	14.57	0.9338	0.9280	0.0058	0.063	0.078	A1Z	.10	Slight tarnish with a clear streak (both phases)
301 SS, Cryoformed	Parent	34	16.27	8.1285	8.1284	0.0001	0.003	0.004	AMX	.10	No apparent reaction
	Welded	34	15.88	8.5774	8.5776	(0.0002)	0	0	AMX	.10	No apparent reaction
	Parent	91	16.03	8.0089	8.0090	(0.0001)	0	0	AMY	.0003	No apparent reaction
	Welded	91	15.80	8.6185	8.6186	(0.0001)	0	0	AMY	.0003	No apparent reaction
	Parent	274	16.14	8.0263	8.0262	0.0001	<.001	<.001	AMZ	.10	No apparent reaction
	Welded	274	16.27	8.8381	8.8381	0	0	0	AMZ	.10	No apparent reaction
303 SS	Parent	34	20.47	24.8956	24.8945	0.0011	0.023	0.028	AMX	.10	Very, very slight stain
	Parent	91	20.47	24.9855	24.9855	0	0	0	AMY	.0003	No apparent reaction
	Parent	274	20.46	25.0760	25.0758	0.0002	0.001	0.001	AMZ	.10	Very slight tarnish
304 SS, Annealed	Parent	34	14.20	1.3668	1.3669	(0.0001)	0	0	AMX	.10	No apparent reaction
	Welded	34	14.23	1.6458	1.6458	0	0	0	AMX	.10	No apparent reaction
	Parent	91	14.38	1.3882	1.3883	(0.0001)	0	0	AMY	.0003	No apparent reaction
	Welded	91	14.11	1.8916	1.8917	(0.0001)	0	0	AMY	.0003	No apparent reaction
	Parent	274	14.29	1.3751	1.3750	0.0001	<.001	<.001	AMZ	.10	No apparent reaction
	Welded	274	14.23	1.9020	1.9016	0.0004	0.001	0.002	AMZ	.10	No apparent reaction

TABLE 2.2-2 (cont.)

Material	Specimen Type	Exposure Time, Days	Specimen Surface Area, cm^2	Specimen Weights, gm Initial	Specimen Weights, gm Final	Specimen Weights, gm Loss	Penetration Rates pm/sec	Penetration Rates mpy	Test No.	Initial Active Fluoride W/O	Observations
304-L SS, Annealed	Parent	34	14.26	0.8455	0.8455	0	0	0	AMX	.10	No apparent reaction
	Welded	34	14.13	1.6230	1.6227	0.0003	0.009	0.011	AMX	.10	No apparent reaction
	Parent	91	13.86	0.8198	0.8199	(0.0001)	0	0	AMY	.0003	Very slight tarnish
	Welded	91	13.87	1.4235	1.4235	0	0	0	AMY	.0003	No apparent reaction
	Parent	274	14.11	0.8353	0.8353	0	0	0	AMZ	.10	No apparent reaction
	Welded	274	14.07	1.8304	1.8310	(0.0007)	0	0	AMZ	.10	No apparent reaction
316-ELC SS, Annealed	Parent	34	14.33	2.0707	2.0707	0	0	0	AMX	.10	No apparent reaction
	Welded	34	14.31	2.8065	2.8067	(0.0002)	0	0	AMX	.10	No apparent reaction
	Parent	91	14.36	2.0885	2.0885	0	0	0	AMY	.0003	No apprent reaction
	Welded	91	14.22	2.7402	2.7402	0	0	0	AMY	.0003	No apparent reaction
	Parent	274	14.24	2.0690	2.0687	0.0003	0.001	0.001	AMZ	.10	No apparent reaction
	Welded	274	14.37	2.7666	2.7659	0.0007	0.003	0.003	AMZ	.10	No apparent reaction
321 SS, Annealed	Parent	34	14.09	2.0223	2.0224	(0.0001)	0	0	AMX	.10	No apparent reaction
	Welded	34	13.90	3.0426	3.0427	(0.0001)	0	0	AMX	.10	No apparent reaction
	Parent	91	14.18	2.0581	2.0581	0	0	0	AMY	.0003	No apparent reaction
	Welded	91	14.06	3.1411	3.1411	0	0	0	AMY	.0003	No apparent reaction
	Parent	274	14.09	2.0449	2.0449	0	0	0	AMZ	.10	Very slight tarnish
	Welded	274	14.11	3.0170	3.0165	0.0005	0.002	0.002	AMZ	.10	Very slight tarnish
347 SS, Annealed	Parent	34	14.29	1.4026	1.4027	(0.0001)	0	0	AMX	.10	No apparent reaction
	Welded	34	14.40	2.3147	2.3147	0	0	0	AMX	.10	No apparent reaction
	Parent	91	14.28	1.4067	1.4067	0	0	0	AMY	.0003	No apparent reaction
	Welded	91	14.46	2.1691	2.1692	(0.0001)	0	0	AMY	.0003	No apparent reaction
	Parent	274	14.35	1.4105	1.4105	0	0	0	AMZ	.10	No apparent reaction
	Welded	274	14.22	2.2720	2.2718	0.0002	0.001	0.001	AMZ	.10	No apparent reaction
A-286 SS	Parent	34	14.51	3.0850	3.0849	0.0001	0.003	0.004	AMX	.10	Very, very slight tarnish
	Parent	91	14.47	3.0538	3.0538	0	0	0	AMY	.0003	Slight tarnish and stain
	Parent	274	14.49	3.0773	3.0762	0.0011	0.004	0.005	AMZ	.10	Very slight stain
17-4PH SS, H-1025	Parent	33	16.23	8.4925	8.4919	0.0006	0.017	0.021	A6X	.0001	Slight stain
	Welded	33	16.14	8.9317	8.9314	0.0003	0.008	0.010	A6X	.0001	Slight stain
	Parent	90	16.26	8.4102	8.4100	0.0002	0.002	0.003	A6Y	.0001	No apparent reaction
	Welded	90	16.03	9.0129	9.0131	(0.0002)	0	0	A6Y	.0001	No apparent reaction
	Parent	269	16.21	8.4401	8.4406	(0.0005)	0	0	A6Z	.10	No apparent reaction
	Welded	269	16.09	8.9545	8.9542	0.0003	0.001	0.001	A6Z	.10	Very slight spotty tarnish in liquid exposure
Nitronic-40 SS	Parent	34	14.56	3.3637	3.3636	0.0001	0.003	0.004	AMX	.10	Very, very slight stain
	Welded	34	14.84	3.7701	3.7699	0.0002	0.006	0.007	AMX	.10	No apparent reaction
	Parent	91	14.57	3.3919	3.3919	0	0	0	AMY	.0003	Slight stain, vapor exposure only
	Welded	91	14.68	3.8115	3.8116	(0.0001)	0	0	AMY	.0003	No apparent reaction
	Parent	274	14.61	3.4079	3.4079	0	0	0	AMZ	.10	Some stain
	Welded	274	14.56	3.7821	3.7819	0.0002	0.001	0.001	AMZ	.10	Some stain

TABLE 2.2-2 (cont.)

Material	Specimen Type	Exposure Time, Days	Specimen Surface Area, cm^2	Specimen Weights, gm Initial	Final	Loss	Penetration Rates pm/sec	mpy	Test No.	Initial Active Fluoride W/O	Observations
Maraging Steel-200	Parent	34	18.04	12.9366	12.9361	0.0005	0.012	0.015	AMX	.10	Slight stain, vapor exposure only
	Welded	34	17.94	13.7126	13.7112	0.0014	0.034	0.042	AMX	.10	Slight stain, vapor exposure only
	Parent	91	17.95	13.0523	13.0523	0	0	0	AMY	.0003	Slight stain
	Welded	91	17.61	13.2834	13.2834	0	0	0	AMY	.0003	Slight stain
	Parent	274	17.88	12.9503	12.9495	0.0008	0.002	0.003	AMZ	.10	Some stain, heavier in vapor exposure
	Welded	274	17.79	13.3778	13.3771	0.0007	0.002	0.003	AMZ	.10	Some stain, heavier in vapor exposure
Maraging Steel-250	Parent	34	16.14	10.8840	10.8830	0.0010	0.027	0.033	AMX	.10	Slight stain, vapor exposure only
	Welded	34	16.34	11.7248	11.7238	0.0010	0.027	0.033	AMX	.10	Slight stain, vapor exposure only
	Parent	91	15.95	11.1841	11.1836	0.0005	0.005	0.006	AMY	.0003	Slight stain
	Welded	91	16.10	10.1742	10.1741	0.0001	0.001	0.001	AMY	.0003	Slight stain
	Parent	274	16.08	10.8850	10.8840	0.0010	0.003	0.004	AMZ	.10	Some stain, heavier in vapor exposure
	Welded	274	16.03	10.7982	10.7972	0.0010	0.003	0.004	AMZ	.10	Some stain, heavier in vapor exposure
Carpenter Custom 455	Parent	34	14.18	2.6160	2.6163	(0.0003)	0	0	AMX	.10	Very slight stain
	Welded	34	13.90	2.6855	2.6854	0.0001	0.003	0.004	AMX	.10	Very slight stain
	Parent	91	14.23	2.6423	2.6420	0.0003	0.003	0.004	A20Y	.0003	Very slight stain
	Welded	91	13.93	2.6603	2.6598	0.0005	0.006	0.007	A20Y	.0003	Very slight stain
	Parent	274	14.15	2.6591	2.6593	(0.0002)	0	0	AMZ	.10	Some stain
	Welded	274	13.84	2.7884	2.7881	0.0003	0.001	0.001	AMZ	.10	Some stain
Inconel-625, Annealed	Parent	34	15.03	3.9761	3.9762	(0.0001)	0	0	AMX	.10	No apparent reaction
	Welded	34	14.84	4.5063	4.5063	0	0	0	AMX	.10	No apparent reaction
	Parent	91	14.99	3.9748	3.9749	(0.0001)	0	0	AMY	.0003	No apparent reaction
	Welded	91	14.76	4.3909	4.3910	(0.0001)	0	0	AMY	.0003	No apparent reaction
	Parent	274	14.88	3.9381	3.9376	0.0005	0.002	0.002	AMZ	.10	No apparent reaction
	Welded	274	14.86	4.5309	4.5305	0.0004	0.001	0.002	AMZ	.10	No apparent reaction
Inconel-718, STA	Parent	34	14.23	1.4144	1.4145	(0.0001)	0	0	AMX	.10	No apparent reaction
	Welded	34	14.19	2.2268	2.2244	0.0024	0.070	0.086	AMX	.10	No apparent reaction
	Parent	91	14.36	3.4624	3.4608	0.0016	0.017	0.021	AMY	.0003	Some stains
	Welded	91	14.83	4.0328	4.0303	0.0025	0.026	0.032	AMY	.0003	Some stains
	Parent	274	14.41	1.4704	1.4702	0.0002	0.001	0.001	AMZ	.10	Some stain
	Welded	274	14.34	1.9935	1.9893	0.0042	0.014	0.018	AMZ	.10	Some stain
Monel-400, Annealed	Parent	34	14.19	2.3049	2.3049	0	0	0	AMX	.10	Slight stain
	Welded	34	13.92	4.0855	4.0855	0	0	0	AMX	.10	Slight stain
	Parent	91	14.17	2.3114	2.3114	0	0	0	AMY	.0003	Some stains
	Welded	91	13.96	4.1055	4.1051	0.0004	0.004	0.005	AMY	.0003	Some stains
	Parent	274	13.98	2.2539	2.2539	0	0	0	AMZ	.10	Light purple stain
	Welded	274	14.39	3.7504	3.7501	0.0003	0.001	0.001	AMZ	.10	Light purple stain

TABLE 2.2-2 (cont.)

Material	Specimen Type	Exposure Time, Days	Specimen Surface Area, cm^2	Specimen Weights, gm Initial	Final	Loss	Penetration Rates pm/sec	mpy	Test No.	Initial Active Fluoride W/O	Observations
Nickel-200, Annealed	Parent	34	14.19	1.5480	1.5479	0.0001	0.003	0.003	AMX	.10	No apparent reaction
	Welded	34	14.15	2.1330	2.1330	0	0	0	AMX	.10	No apparent reaction
	Parent	91	14.31	1.5617	1.5618	(0.0001)	0	0	AMY	.0003	Tarnish and stains
	Welded	91	14.24	2.1744	2.1745	(0.0001)	0	0	AMY	.0003	Slight tarnish
	Parent	274	14.22	1.5438	1.5437	0.0001	<.001	<.001	AMZ	.10	Slight tarnish
	Welded	274	14.20	2.1958	2.1950	0.0008	0.003	0.003	AMZ	.10	Slight tarnish
Nickel-270, Annealed	Parent	34	17.66	16.4182	16.4182	0	0	0	AMX	.10	No apparent reaction
	Welded	34	17.85	17.3177	17.3177	0	0	0	AMX	.10	No apparent reaction
	Parent	91	17.73	16.2625	16.2621	0.0004	0.003	0.004	AMY	.0003	Slight stain
	Welded	91	18.01	17.3377	17.3373	0.0004	0.003	0.004	AMY	.0003	Slight stain
	Parent	274	17.54	16.2995	16.2982	0.0013	0.004	0.004	AMZ	.10	Slight tarnish
	Welded	274	18.06	17.6850	17.6836	0.0014	0.004	0.005	AMZ	.10	Slight tarnish
Titanium 6Al-4V, STA	Parent	33	15.12	2.4316	2.4316	0	0	0	A2X	.0001	Very slight stain
	Welded	33	15.06	2.7464	2.7466	(0.0002)	0	0	A2X	.0001	Very slight stain
	Parent	90	14.65	2.0449	2.0448	0.0001	0.002	0.002	A2Y	.0001	No apparent reaction
	Welded	90	14.58	2.2619	2.2617	0.0002	0.004	0.005	A2Y	.0001	No apparent reaction
	Parent	271	15.15	2.4434	2.4425	0.0009	0.006	0.007	A2Z	.10	Very slight purplish stain
	Welded	271	14.92	2.7668	2.7661	0.0007	0.004	0.006	A2Z	.10	Very slight purplish stain
Titanium 5Al-2.5 Sn, ELI	Parent	33	14.88	2.3581	2.3581	0	0	0	A2X	.0001	Very slight stain
	Welded	33	14.84	2.6215	2.6215	0	0	0	A2X	.0001	Very slight stain
	Parent	90	15.17	2.3980	2.3978	0.0002	0.004	0.005	A2Y	.0001	No apparent reaction
	Welded	90	14.99	2.6873	2.6871	0.0002	0.004	0.005	A2Y	.0001	Slight purplish tinge on edge
	Parent	271	15.01	2.3686	2.3678	0.0008	0.005	0.006	A2Z	.10	Very slight purplish stain
	Welded	271	14.87	2.6866	2.6859	0.0007	0.004	0.005	A2Z	.10	Very slight purplish stain
Copper, OFHC	Parent	34	14.28	1.5624	1.5601	0.0023	0.060	0.074	AMX	.10	Some corrosion, greater in liquid exposure
	Welded	34	14.46	3.5182	3.5159	0.0023	0.059	0.073	AMX	.10	Some corrosion, greater in liquid exposure
	Parent	91	14.22	1.5561	1.5561	0	0	0	AMY	.0003	Slight tarnish
	Welded	91	14.29	3.1560	3.1557	0.0003	0.003	0.004	AMY	.0003	Slight tarnish
	Parent	274	14.03	1.5370	1.5275	0.0095	0.032	0.040	AMZ	.10	Some tarnish, white deposit in liquid exposure
	Welded	274	14.31	3.4411	3.4282	0.0129	0.043	0.053	AMZ	.10	Some tarnish, white deposit in liquid exposure
Beryllium Copper	Parent	33	14.25	1.4807	1.4794	0.0013	0.039	0.048	A5X	.10	Slight tarnish
	Parent	90	14.16	1.4793	1.4789	0.0004	0.004	0.005	A5Y	.0001	No apparent reaction
	Parent	269	14.30	1.4868	1.4853	0.0015	0.005	0.006	A5Z	.10	Tarnished
Aluminum-Bronze 623	Parent	33	17.97	16.9443	16.9437	0.0006	0.015	0.019	A3X	.0001	Very slight tarnish
	Parent	90	17.94	16.8973	16.8948	0.0025	0.023	0.029	A3Y	.0001	Light stain over entire surface
	Parent	270	17.96	16.9737	16.9718	0.0019	0.006	0.007	A3Z	.10	Light stain, heavier in liquid exposure
Tungsten-2% Th	Parent	33	8.78	12.4464	12.4464	0	0	0	A4X	.0001	No apparent reaction
	Parent	90	8.61	12.4373	12.4371	0.0002	0.002	0.003	A4Y	.0001	No apparent reaction
	Parent	270	8.92	12.8788	12.8782	0.0006	0.001	0.002	A4Z	.10	Gray color slightly darker in liquid exposure

TABLE 2.2-3

DATA INDICATIVE OF THE COMPATIBILITY OF VAPOR PHASE NITROGEN TRIFLUORIDE AT 344 K (160°F) AND 3.45 MN/m^2 (500 PSIA) WITH VARIOUS METALS

Material	Specimen Type	Exposure Time, Days	Specimen Surface Area, cm^2	Specimen Weights, gm Initial	Specimen Weights, gm Final	Specimen Weights, gm Loss	Penetration Rates pm/sec	Penetration Rates mpy	Test No.	Initial Active Fluoride W/O	Observations
1100 Aluminum	Parent	32	14.36	0.4954	0.4953	0.0001	0.009	0.011	B1X	.0001	No apparent reaction
	Welded	32	14.25	0.7814	0.7813	0.0001	0.009	0.012	B1X	.0001	No apparent reaction
	Parent	90	14.44	0.4963	0.4962	0.0001	0.003	0.004	B1Y	.0001	Slight tarnish
	Welded	90	14.36	0.7293	0.7292	0.0001	0.003	0.004	B1Y	.0001	Slight tarnish
	Parent	271	14.25	0.4894	0.4852	0.0042	0.047	0.057	B1Z	.10	Slight tarnish
	Welded	271	14.28	0.7255	0.7214	0.0041	0.045	0.056	B1Z	.10	Slight tarnish
2014, T6 Aluminum	Parent	32	16.36	4.0673	4.0569	0.0004	0.033	0.04[illegible]	B1X	.0001	Very slight stain
	Welded	32	16.45	4.3765	4.3763	0.0003	0.024	0.030	B1X	.0001	Very slight stain
	Parent	90	16.53	4.1198	4.1197	0.0001	0.003	0.004	B1Y	.0001	No apparent reaction
	Welded	90	16.41	4.3439	4.3438	0.0001	0.003	0.004	B1Y	.0001	No apparent reaction
	Parent	271	16.47	4.0966	4.0956	0.0010	0.010	0.012	B1Z	.10	Slight tarnish
	Welded	271	16.57	4.3475	4.3455	0.0020	0.019	0.024	B1Z	.10	Slight tarnish
2219, T87 Aluminum	Parent	32	14.95	1.6889	1.6087	0.0002	0.018	0.022	B1X	.0001	Very slight stain
	Welded	32	14.99	2.0099	2.0095	0.0004	0.036	0.044	B1X	.0001	Very slight stain
	Parent	90	14.97	3.5519	3.5518	0.0001	0.003	0.004	B1Y	.0001	No apparent reaction
	Welded	90	14.80	3.7388	3.7386	0.0002	0.006	0.008	B1Y	.0001	No apparent reaction
	Parent	271	14.95	1.6255	1.6247	0.0008	0.008	0.010	B1Z	.10	Slight tarnish
	Welded	271	14.84	1.9804	1.9783	0.0021	0.022	0.028	B1Z	.10	Slight tarnish
6061, T6 Aluminum	Parent	32	14.59	0.4779	0.4779	0	0	0	B1X	.0001	Trace of slight stain
	Welded	32	14.64	2.8467	0.8449	0.0018	0.164	0.204	B1X	.[illegible]01	Trace of slight stain
	Parent	90	14.12	0.4692	0.4694	(0.0002)	0	0	B1Y	.0001	Slight tarnish
	Welded	90	14.74	0.8421	0.8408	0.0013	0.042	0.052	B1Y	.0001	Slight tarnish
	Parent	271	14.25	0.4637	0.4602	0.0035	0.039	0.048	B1Z	.10	Slight tarnish
	Welded	271	14.71	0.9798	0.9685	0.0113	0.121	0.151	B1Z	.10	Slight tarnish
301 SS, Cryoformed	Parent	30	15.89	7.6776	7.6775	0.0001	0.003	0.004	BMX	.10	Purple stain
	Welded	30	16.49	8.8430	8.8429	0.0001	0.003	0.[illegible]	BMX	.10	Purple stain concentrated at weld
	Parent	91	16.09	8.1104	8.1101	0.0003	0.003	0.004	BMY	.0001	Light purple stain
	Welded	91	15.85	8.5007	8.5004	0.0003	0.003	0.004	BMY	.0001	Light purple stain
	Parent	270	16.19	8,0803	8.0805	(0.0002)	0	0	BMZ	.10	Heavy brown and purple stain
	Welded	270	16.00	8.6719	8.6723	(0.0005)	0	0	BMZ	.10	Heavy brown and purple stain
303 SS	Parent	30	20.32	24.6198	24.6189	0.0009	0.021	0.026	BMX	.10	Slight purple stain
	Parent	91	20.42	24.9400	24.9389	0.0011	0.009	0.011	BMY	.000[illegible]	Slight tarnish
	Parent	270	20.29	24.6384	24.6373	0.0011	0.003	0.004	BMZ	.10	Light brown and purple stain
304 SS, Annealed	Parent	30	14.12	1.3571	1.3573	(0.0002)	0	0	BMX	.10	Slight tarnish
	Welded	30	14.30	1.9656	1.9656	0	0	0	BMX	.10	Slight purple stain
	Parent	91	14.23	1.3681	1.3679	0.0002	0.002	0.003	BMY	.0003	Very slight purple stain
	Welded	91	14.05	2.0548	2.0545	0.0003	0.003	0.004	BMY	.0003	Very slight purple stain
	Parent	270	14.33	1.3850	1.3847	0.0003	0.001	0.001	BMZ	.10	Some purple and yellow stain
	Welded	270	14.30	1.7631	1.7628	0.0003	0.001	0.001	BMZ	.10	Some purple and yellow stain

TABLE 2.2-3 (cont.)

Material	Specimen Type	Exposure Time, Days	Specimen Surface Area, cm^2	Specimen Weights, gm Initial	Final	Loss	Penetration Rates pm/sec	mpy	Test No.	Initial Active Fluoride W/O	Observations
304-L SS, Annealed	Parent	30	14.26	0.8493	0.8444	0.0049	0.164	0.205	BMX	.10	Very slight purple stain
	Welded	30	14.17	1.0415	1.0414	0.0001	0.003	0.004	BMX	.10	Purple stain
	Parent	91	14.05	0.8349	0.8348	0.0001	0.001	0.001	BMY	.0003	Very slight purple stain
	Welded	91	14.03	1.1204	1.1201	0.0003	0.003	0.004	BMY	.0003	Very slight purple stain
	Parent	270	14.11	0.8414	0.8418	(0.0004)	0	0	BMZ	.10	Some purple and yellow stain
	Welded	270	13.88	1.2312	1.2305	0.0007	0.003	0.003	BMZ	.10	Some purple and yellow stain
316-ELC SS, Annealed	Parent	30	14.27	2.0718	2.0717	0.0001	0.003	0.004	BMX	.10	No apparent reaction
	Welded	30	14.34	2.8689	2.8689	0	0	0	BMX	.10	No apparent reaction
	Parent	91	14.24	2.0569	2.0568	0.0001	0.001	0.001	BMY	.0001	No apparent reaction
	Welded	91	14.11	2.9184	2.9181	0.0003	0.003	0.004	BMY	.0001	No apparent reaction
	Parent	270	14.09	2.0017	2.0015	0.0002	0.001	0.001	BMZ	.10	Some purple and yellow stain
	Welded	270	14.53	2.7248	2.7246	0.0002	0.001	0.001	BMZ	.10	Some purple and yellow stain
321 SS, Annealed	Parent	30	14.26	2.0781	2.0780	0.0001	0.003	0.004	BMX	.10	Slight purple stain
	Welded	30	14.40	3.2566	3.2564	0.0002	0.007	0.008	BMX	.10	Slight purple stain
	Parent	91	14.31	2.0473	2.0471	0.0002	0.002	0.003	BMY	.0003	Light purple stains
	Welded	91	13.84	2.8484	2.8481	0.0003	0.003	0.004	BMY	.0003	Light purple stains
	Parent	270	14.25	2.0676	2.0673	0.0003	0.001	0.001	BMZ	.10	Some purple and yellow stain
	Welded	270	14.20	2.8654	2.8647	0.0007	0.003	0.003	BMZ	.10	Some purple and yellow stain
347-SS, Annealed	Parent	30	14.42	1.4062	1.4061	0.0001	0.003	0.004	BMX	.10	Slight purple stain
	Welded	30	14.29	2.1573	2.1570	0.0003	0.010	0.013	BMX	.10	Slight purple stain
	Parent	91	14.36	1.4016	1.4012	0.0004	0.004	0.005	BMY	.0003	Light purple stains
	Welded	91	14.25	2.5287	2.5282	0.0005	0.006	0.007	BMY	.0003	Light purple stains
	Parent	270	14.34	1.4056	1.4052	0.0004	0.001	0.002	BMZ	.10	Some purple and yellow stain
	Welded	270	14.42	2.0807	2.0800	0.0007	0.002	0.003	BMZ	.10	Some purple and yellow stain
A-286 SS	Parent	30	14.60	3.0938	3.0935	0.0003	0.010	0.012	BMX	.10	Slight purple stain
	Parent	91	14.50	3.0667	3.0663	0.0004	0.004	0.005	BMY	.0003	Dark gray stains
	Parent	270	14.48	3.0722	3.0710	0.0012	0.004	0.006	BMZ	.10	Very light brown and purple stain
17-4 PH SS, H-1025	Parent	32	16.24	8.4524	8.4444	0.0080	0.229	0.285	B6X	.0001	Dark brown deposit
	Welded	32	15.78	8.7704	8.7640	0.0064	0.189	0.235	B6X	.0001	Dark brown deposit
	Parent	90	15.67	8.4782	8.4727	0.0055	0.058	0.072	B6Y	.0001	Black coating on surface
	Welded	90	15.61	8.8328	8.8320	0.0008	0.008	0.011	B6Y	.0001	Black spots on surface
	Parent	271	16.22	8.3686	8.3518	0.0168	0.055	0.069	B6Z	.10	Dark brown coating
	Welded	271	16.19	9.0330	9.0233	0.0097	0.032	0.040	B6Z	.10	Dark brown coating
Nitronic-40 SS	Parent	30	14.37	3.3447	3.3445	0.0002	0.007	0.008	BMX	.10	No apparent reaction
	Welded	30	14.53	3.8137	3.8136	0.0001	0.003	0.004	BMX	.10	Slight purple stain on one edge
	Parent	91	14.44	3.3441	3.3439	0.0002	0.002	0.003	BMY	.0003	Very light tarnish
	Welded	91	14.68	3.8376	3.8374	0.0002	0.002	0.003	BMY	.0003	Very light tarnish
	Parent	270	14.60	3.3993	3.3987	0.0006	0.002	0.003	BMZ	.10	Heavy brown and purple stain
	Welded	270	14.96	3.7906	3.7900	0.0006	0.002	0.003	BMZ	.10	Heavy brown and purple stain

TABLE 2.2-3 (cont.)

Material	Specimen Type	Exposure Time, Days	Specimen Surface Area, cm^2	Specimen Weights, gm Initial	Final	Loss	Penetration Rates pm/sec	mpy	Test No.	Initial Active Fluoride W/O	Observations
Maraging Steel-200	Parent	30	18.09	13.1134	13.1124	0.0010	0.027	0.034	BMX	.10	Purple stain
	Welded	30	17.81	13.1797	13.1787	0.0010	0.028	0.034	BMX	.10	Purple stain
	Parent	91	17.77	12.9075	12.9057	0.0018	0.016	0.020	BMY	.0003	Dark gray stains
	Welded	91	17.84	13.5349	13.5331	0.0018	0.016	0.020	BMY	.0003	Dark gray stains
	Parent	270	17.92	12.9956	12.9885	0.0071	0.021	0.026	BMZ	.10	Heavy dark gray stain
	Welded	270	17.97	13.6916	13.6882	0.0034	0.010	0.013	BMZ	.10	Heavy dark gray stain
Maraging Steel-250	Parent	30	16.52	10.9744	10.9729	0.0013	0.039	0.048	BMX	.10	Purple stain
	Welded	30	16.17	11.6276	11.6268	0.0008	0.024	0.030	BMX	.10	Purple stain
	Parent	91	15.33	8.7766	8.7755	0.0011	0.012	0.014	BMY	.0003	Dark gray stains
	Welded	91	15.91	10.1724	10.1712	0.0012	0.012	0.015	BMY	.0003	Dark gray stains
	Parent	270	16.40	11.2974	11.2923	0.0051	0.017	0.021	BMZ	.10	Heavy dark gray stain
	Welded	270	16.23	11.5914	11.5884	0.0030	0.010	0.012	BMZ	.10	Heavy dark gray stain
Steel, C1010	Parent	32	13.97	1.3312	1.3302	0.0010	0.033	0.041	B7X	.0001	Some purple stain
	Welded	32	14.07	1.6279	9.6259	0.0020	0.065	0.081	B7X	.0001	Some purple stain
	Parent	90	13.95	1.3270	1.3262	0.0008	0.009	0.012	B7Y	.0001	Slight reddish tarnish
	Welded	90	13.91	1.6005	1.5988	0.0017	0.020	0.025	B7Y	.0001	Slight reddish tarnish
	Parent	271	13.98	1.3246	1.3228	0.0018	0.007	0.009	B7Z	.10	Light purple stain
	Welded	271	14.06	1.6669	1.6636	0.0033	0.013	0.016	B7Z	.10	Light purple stain
Carpenter Custom 455	Parent	30	14.17	2.6274	2.5938	0.0336	1.14	1.42	BMX	.10	Heavy black deposit
	Welded	30	13.88	2.6020	2.5728	0.0292	1.01	1.26	BMX	.10	Heavy black deposit
	Parent	91	14.23	2.6222	2.6197	0.0025	0.028	0.035	B20Y	.0003	Stained, brown and gray
	Welded	91	13.81	2.8874	2.8853	0.0021	0.024	0.030	B20Y	.0003	Stained, brown and gray
	Parent	270	14.14	2.6263	2.5355	0.0908	0.343	0.427	BMZ	.10	Heavy brown and black deposit
	Welded	270	13.79	2.6028	2.5148	0.0880	0.341	0.424	BMZ	.10	Heavy brown and black deposit
Inconel-625, Annealed	Parent	30	14.86	3.9187	3.9188	(0.0001)	0	0	BMX	.10	No apparent reaction
	Welded	30	14.85	4.4505	4.4504	0.0001	0.003	0.004	BMX	.10	No apparent reaction
	Parent	91	14.93	3.9604	3.9603	0.0001	0.001	0.001	BMY	.0003	No apparent reaction
	Welded	91	14.75	4.2587	4.2587	0	0	0	BMY	.0003	No apparent reaction
	Parent	270	14.91	3.9462	3.9460	0.0002	0.001	0.001	BMZ	.10	Very light tarnish
	Welded	270	14.87	4.4127	4.4125	0.0002	0.001	0.001	BMZ	.10	Very light tarnish
Inconel-718, STA	Parent	30	14.18	1.4068	1.4067	0.0001	0.003	0.004	BMX	.10	No apparent reaction
	Welded	30	14.18	1.9295	1.9278	0.0017	0.056	0.070	BMX	.10	No apparent reaction
	Parent	91	14.36	3.4967	3.4949	0.0018	0.019	0.024	BMY	.0003	Some black stain
	Welded	91	14.60	4.0214	4.0128	0.0086	0.091	0.113	BMY	.0003	Some black stain
	Parent	270	14.13	1.4072	1.4069	0.0003	0.001	0.001	BMZ	.10	Heavy black stain
	Welded	270	14.34	1.9701	1.9617	0.0084	0.030	0.038	BMZ	.10	Heavy black stain

TABLE 2.2-3 (cont.)

Material	Specimen Type	Exposure Time, Days	Specimen Surface Area, cm^2	Specimen Weights, gm Initial	Final	Loss	Penetration Rates pm/sec	mpy	Test No.	Initial Active Fluoride W/O	Observations
Monel-400, Annealed	Parent	30	14.24	2.3178	2.3179	(0.0001)	0	0	BMX	.10	No apparent reaction
	Welded	30	14.14	4.1419	4.1418	0.0001	0.003	0.004	BMX	.10	No apparent reaction
	Parent	91	14.21	2.3148	2.3147	0.0001	0.001	0.001	BMY	.0003	Very light tarnish
	Welded	91	13.95	3.6318	3.6316	0.0002	0.002	0.003	BMY	.0003	Very light tarnish
	Parent	270	14.14	2.3078	2.3078	0	0	0	BMZ	.10	Very light tarnish
	Welded	270	14.42	3.8745	3.8740	0.0005	0.002	0.002	BMZ	.10	Very light tarnish
Nickel-200, Annealed	Parent	30	14.21	1.5384	1.5383	0.0001	0.003	0.004	BMX	.10	No apparent reaction
	Welded	30	14.26	2.3529	2.3528	0.0001	0.003	0.004	BMX	.10	No apparent reaction
	Parent	91	14.28	1.5482	1.5481	0.0001	0.001	0.001	BMY	.0003	Very light tarnish
	Welded	91	14.24	2.1845	2.1844	0.0001	0.002	0.003	BMY	.0003	Very light tarnish
	Parent	270	14.22	1.5470	1.5469	0.0001	<0.001	<0.001	BMZ	.10	Some tarnish
	Welded	270	14.15	2.1896	2.1892	0.0004	0.001	0.002	BMZ	.10	Some tarnish
Nickel-270, Annealed	Parent	30	17.68	16.3039	16.3037	0.0002	0.005	0.006	BMX	.10	No apparent reaction
	Welded	30	17.96	17.6553	17.6552	0.0001	0.002	0.003	BMX	.10	No apparent reaction
	Parent	91	17.60	16.3774	16.3766	0.0008	0.006	0.008	BMY	.0003	Very light tarnish
	Welded	91	17.89	17.1142	17.1138	0.0004	0.003	0.004	BMY	.0003	Very light tarnish
	Parent	270	17.76	16.4769	16.4761	0.0008	0.002	0.003	BMZ	.10	Very light tarnish
	Welded	270	17.92	17.9026	17.9007	0.0019	0.005	0.006	BMZ	.10	Very light tarnish
Titanium 6Aℓ-4V, STA	Parent	32	15.17	2.4540	2.4539	0.0001	0.005	0.007	B2X	.0001	Some stain
	Welded	32	15.03	3.0847	3.0837	0.0010	0.054	0.067	B2X	.0001	Some stain
	Parent	90	14.67	2.0159	2.0153	0.0006	0.012	0.014	B2Y	.0001	Light film, purple and gray coloration
	Welded	90	15.02	2.5978	2.5972	0.0006	0.011	0.014	B2Y	.0001	Light film, purple and gray coloration
	Parent	271	15.13	2.4601	2.4559	0.0042	0.027	0.033	B2Z	.10	Light gray, very hygroscopic film
	Welded	271	14.95	2.7617	2.7585	0.0032	0.020	0.025	B2Z	.10	Light gray, very hygroscopic film
Titanium 4Aℓ-2.5 Sn, ELI	Parent	32	14.98	2.3727	2.3708	0.0019	0.103	0.128	B2X	.0001	Some stain
	Welded	32	14.97	2.7753	2.7735	0.0018	0.097	0.121	B2X	.0001	Some stain
	Parent	90	15.11	2.3990	2.3972	0.0018	0.034	0.042	B2Y	.0001	Dark gray film covering specimen
	Welded	90	14.95	2.6940	2.6921	0.0019	0.036	0.045	B2Y	.0001	Dark gray film covering specimen
	Parent	271	14.91	2.3478	2.3388	0.0090	0.057	0.071	B2Z	.10	Gray, very hygroscopic stain, turning
	Welded	271	14.88	2.7340	2.7247	0.0093	0.059	0.074	B2Z	.10	brown after exposure to air
Copper, OFHC	Parent	30	14.27	1.5630	1.5629	0.0001	0.003	0.004	BMX	.10	Very slight tarnish
	Welded	30	14.23	3.5280	3.5277	0.0003	0.009	0.011	BMX	.10	No apparent reaction
	Parent	91	14.30	1.5671	1.5663	0.0008	0.008	0.010	BMY	.0003	Very light tarnish
	Welded	91	14.25	3.6391	3.6378	0.0013	0.013	0.016	BMY	.0003	Very light tarnish
	Parent	270	14.32	1.5708	1.5706	0.0002	0.001	0.001	BMZ	.10	Very light tarnish
	Welded	270	14.20	3.3371	3.3364	0.0007	0.002	0.003	BMZ	.10	Very light tarnish

TABLE 2.2-3 (cont.)

Material	Specimen Type	Exposure Time, Days	Specimen Surface Area, cm^2	Specimen Weights, gm Initial	Final	Loss	Penetration Rates pm/sec	mpy	Test No.	Initial Active Fluoride W/O	Observations
Beryllium Copper	Parent	33	14.19	1.4795	1.4782	0.0013	0.039	0.048	B5X	.0001	Slight tarnish
	Parent	90	14.24	1.4774	1.4762	0.0012	0.013	0.016	B5Y	.0001	Tarnished surface
	Parent	270	14.34	1.4840	1.4840	0	0	0	B5Z	.10	Some tarnish
Aluminum Bronze-623	Parent	32	17.91	16.7817	16.7806	0.0011	0.029	0.035	B3X	.0001	Slight tarnish
	Parent	90	17.97	16.8248	16.8232	0.0016	0.015	0.018	B3Y	.0001	Surface film covering specimen
	Parent	270	17.89	16.7839	16.7829	0.0010	0.003	0.004	B3Z	.10	Covered with a brown stain
Tungsten-2% Tn	Parent	33	8.72	12.6088	12.6052	0.0036	0.075	0.093	B4X	.0001	Stain gray to dark gray
	Parent	90	8.55	12.4009	12.4011	(0.0002)	0	0	B4Y	.0001	Dark gray film covering specimens
	Parent	270	8.87	12.7110	12.5708	0.1402	0.350	0.435	B4Z	.10	Covered with a very hygroscopic powder. Turned gray after hydrolyzing

6061-T-6 aluminum (0.12 pm/sec, 0.15 mpy), and tungsten (0.35 pm/sec, 0.44 mpy) exhibited any significant corrosion, that is, a rate greater than 0.08 pm/sec (0.10 mpy); (2) the aluminum alloys corrosion penetration rates ranged from 0.01 pm/sec (0.012 mpy) to 0.12 pm/sec (0.15 mpy); and the 300 series stainless steel alloys and nickel exhibited rates equal to or less than 0.005 pm/sec (0.006 mpy); and the rates for the titanium alloys ranged from 0.02 pm/sec (0.025 mpy) to 0.059 pm/sec (0.074 mpy). The nitrogen trifluoride used for the 270 day exposure tests contained 0.10 weight percent active fluoride calculated as hydrogen fluoride while all the 90 day exposure tests reported in Table 2.2-3 were conducted with nitrogen trifluoride which contained 0.0003 weight percent active fluoride or less. For the 30 day exposure tests labeled BMX the nitrogen trifluoride contained 0.10 weight percent active fluoride while all the other 30 day exposure tests were conducted with nitrogen trifluoride which initially contained 0.0001 weight percent active fluoride. During the 270 day exposure tests the oven which contained the sample containers did undergo a temperature excursion from 344 K (160 F) to 422 K (300 F) for a two day period during a weekend. An examination of the test data indicates that the event did not adversely affect the metal corrosion rates.

The experimental results obtained from the gaseous static exposure tests with selected metals for duration of 30, 90 and 270 days at 344 K (160 F) and pressures up to 17.24 MN/m^2 (2500 psia) are presented in Table 2.2-4. The significant items to note from the data are as follows: (1) generally the corrosion rate, based on 270 day data, increases slightly with pressure but the rates for all the metals tested are very low, the highest measured rate is 0.18 pm/sec (0.22 mpy), (2) the corrosion which occurs apparently does so during the initial portion of the exposure period, the 30 day rates are generally greater than the 270 day rates, and (3) the chemical compatibility of the metals selected for testing with nitrogen trifluoride increases in the order Maraging steel 200, Maraging steel 250, titanium 6Al-4V, Inconel 718 STA, 2219 aluminum, T-87, 1010 carbon steel and CRES 301, cryoformed.

The chemical analyses of the nitrogen trifluoride recovered from the tests are presented in Table 2.2-5. The chemical analysis of the nitrogen trifluoride as it was received in the cylinders which were used to fill the test containers are included in the table for the reader's convenience. The data in the table indicate that no significant decomposition of nitrogen trifluoride occurred in the presence of the metals in the liquid/vapor tests at 195 K (-78 C). At 344 K (160 F) and at pressures from 3.45 MN/m^2 (500 psia) to 17.24 MN/m^2 (2500 psia) the nitrogen trifluoride did not decompose to a significant extent although there is some evidence that there is a nitrogen-forming decomposition reaction occurring at the rate of a few tenths of a percent per year.

TABLE 2.2-4

DATA INDICATIVE OF THE COMPATIBILITY OF VAPOR PHASE NITROGEN TRIFLUORIDE AT 344 K (160 F) AND PRESSURES GREATER THAN 3.45 MN/m2 (500 PSIA) WITH VARIOUS METALS

Material	Specimen Type	Exposure Time, days	Test Pressure MN/m^2	Test Pressure psia	Specimen Surface Area cm^2	Specimen Weights, gm Initial	Specimen Weights, gm Final	Specimen Weights, gm Loss	Penetration Rates pm/sec	Penetration Rates mpy	Test No.	Initial Active Fluoride Content Weight %	Observations
2219, T87 Aluminum	Parent	32	8.62	1250	15.44	1.6953	1.6949	0.0004	0.035	0.043	C1X	.0001	No apparent reaction
	Welded	32	8.62	1250	15.23	4.1626	4.1620	0.0006	0.053	0.065	C1X	.0001	No apparent reaction
	Parent	32	13.44	1950	15.12	3.5818	3.5813	0.0005	0.044	0.055	D1X	.001	No apparent reaction
	Welded	32	13.44	1950	15.15	4.0581	4.0576	0.0005	0.044	0.055	D1X	.001	No apparent reaction
	Parent	87	10.34	1500	14.91	3.5556	3.5554	0.0002	0.007	0.008	C1Y	.0002	No apparent reaction
	Welded	87	10.34	1500	14.90	3.8562	3.85[illegible]	0.0004	0.013	0.016	C1Y	.0002	No apparent reaction
	Parent	273	10.34	1500	15.32	1.6595	1.6586	0.0009	0.009	0.011	C1Z	.0002	Slight tarnish
	Welded	273	10.34	1500	15.17	2.0601	2.0553	0.0048	0.050	0.062	C1Z	.0002	Slight tarnish
	Parent	32	17.24	2500	15.12	3.5818	3.5806	0.0012	0.11	0.13	*D1X	.0003	No apparent reaction
	Welded	32	17.24	2500	15.15	4.0581	4.0569	0.0012	0.11	0.13	*D1X	.0003	No apparent reaction
	Parent	87	17.24	2500	15.03	3.5808	3.5798	0.0010	0.033	0.041	D1Y	.0002	No apparent reaction
	Welded	87	17.24	2500	15.33	4.1568	4.1525	0.0043	0.138	0.171	D1Y	.0002	No apparent reaction
	Parent	269	17.24	2500	14.90	1.6388	1.6371	0.0017	0.018	0.023	D1Z	.0002	Very light tarnish, some stain spots
	Welded	269	17.24	2500	15.09	1.8655	1.8620	0.0035	0.037	0.046	D1Z	.0002	Very light tarnish, some stain spots
301 SS, Cryoformed	Parent	30	8.62	1250	16.06	7.9857	7.9859	(0.0002)	-0-	-0-	CMX	.17	Purple stain
	Welded	30	8.62	1250	15.91	8.5588	8.5588	-0-	-0-	-0-	CMX	.17	Purple stain
	Parent	30	13.44	1950	16.13	8.0977	8.0978	(0.0001)	-0-	-0-	DMX	.17	Slight purple stain
	Welded	30	13.44	1950	15.94	8.5549	8.5550	(0.0001)	-0-	-0-	DMX	.17	Slight purple stain
	Parent	91	10.34	1500	16.26	8.1713	8.1712	0.0001	0.001	0.001	CMY	.0003	Some purple stain
	Welded	91	10.34	1500	15.72	8.3340	8.3334	0.0006	0.006	0.008	CMY	.0003	Some purple stain
	Parent	269	10.34	1500	16.08	8.0751	8.0744	0.0007	0.002	0.003	CMZ	.14	Light purple stain
	Welded	269	10.34	1500	16.21	8.8191	8.8184	0.0007	0.002	0.003	CMZ	.14	Light purple stain
	Parent	29	17.24	2500	16.13	8.0977	8.0973	0.0004	0.012	0.015	*DMX	.0002	Purple-gray stain
	Welded	29	17.24	2500	15.94	8.5549	8.5536	0.0013	0.041	0.050	*DMX	.0002	Purple-gray stains
	Parent	92	17.24	2500	16.16	8.0106	8.0102	0.0004	0.004	0.005	DMY	.0003	Purple stain
	Welded	92	17.24	2500	16.16	8.6184	8.6176	0.0008	0.008	0.010	DMY	.0003	Purple stain
	Parent	270	17.24	2500	16.05	8.0657	8.0648	0.0009	0.003	0.004	DMZ	.13	Purple stain
	Welded	270	17.24	2500	16.41	8.9194	8.9184	0.0010	0.003	0.004	DMZ	.13	Purple stain
Maraging Steel-200	Parent	30	8.62	1250	18.19	13.2202	13.2157	0.0045	0.121	0.151	CMX	.17	Slight amber stain around holes
	Welded	30	8.62	1250	17.79	13.4976	13.4952	0.0024	0.066	0.082	CMX	.17	Slight amber stain around holes
	Parent	30	13.44	1950	18.02	13.0927	13.0904	0.0023	0.063	0.078	DMX	.17	Rust colored stain around holes
	Welded	30	13.44	1950	17.98	13.5979	13.5957	0.0022	0.060	0.074	DMX	.17	Rust colored stain around holes
	Parent	91	10.34	1500	17.75	12.7984	12.7834	0.0150	0.137	0.170	CMY	.0003	Some gray stain
	Welded	91	10.34	1500	16.95	13.6997	13.6890	0.0107	0.102	0.127	CMY	.0003	Some gray stain
	Parent	269	10.34	1500	17.91	12.9986	12.9653	0.0333	0.102	0.126	CMZ	.14	Dark gray film
	Welded	269	10.34	1500	17.82	13.4632	13.4068	0.0564	0.173	0.215	CMZ	.14	Dark gray film
	Parent	29	17.24	2500	18.02	13.0927	13.0837	0.0090	0.254	0.315	*DMX	.0002	Light gray to black coating
	Welded	29	17.24	2500	17.98	13.5979	13.5911	0.0068	0.192	0.230	*DMX	.0002	Light gray to black coating
	Parent	92	17.24	2500	17.86	12.9807	12.9628	0.0179	0.160	0.199	DMY	.0003	Purplish-gray coating
	Welded	92	17.24	2500	17.92	13.3318	13.3225	0.0093	0.084	0.104	DMY	.0003	Purplish-gray coating
	Parent	270	17.24	2500	17.85	12.9106	12.8556	0.0550	0.168	0.208	DMZ	.13	Gray-brown coating
	Welded	270	17.24	2500	17.92	13.7313	13.6750	0.0563	0.171	0.213	DMZ	.13	Gray-brown coating

TABLE 2.2-4 (cont.)

Material	Specimen Type	Exposure Time, days	Test Pressure MN/m^2	Test Pressure psia	Specimen Surface Area cm^2	Specimen Weights, gm Initial	Specimen Weights, gm Final	Specimen Weights, gm Loss	Penetration Rates pm/sec	Penetration Rates mpy	Test No.	Initial Active Fluoride Content Weight %	Observations
Maraging Steel-250	Parent	30	8.62	1250	16.05	10.1023	10.0978	0.0045	0.137	0.170	CMX	.17	Slight amber stain around holes
	Welded	30	8.62	1250	16.06	11.6649	11.6609	0.0040	0.137	0.170	CMX	.17	Slight amber stain around holes
	Parent	30	13.44	1950	16.31	11.2904	11.2871	0.0033	0.099	0.123	DMX	.17	Rust-colored stain around holes
	Welded	30	13.44	1950	15.87	10.6223	10.6916	0.0027	0.083	0.104	DMX	.17	Rust-colored stain around holes
	Parent	91	10.34	1500	15.85	8.8215	8.8118	0.0097	0.099	0.123	CMY	.0003	Some gray stain
	Welded	91	10.34	1500	16.18	11.3398	11.3309	0.0089	0.089	0.110	CMY	.0003	Some gray stain
	Parent	269	10.34	1500	16.05	10.4585	10.4308	0.0277	0.094	0.117	CMZ	.14	Dark gray film
	Welded	269	10.34	1500	15.73	10.0943	10.0644	0.0299	0.104	0.129	CMZ	.14	Dark gray film
	Parent	29	17.24	2500	16.31	11.2904	11.2793	0.0111	0.345	0.429	*DMX	.0002	Light gray to black coating
	Welded	29	17.24	2500	15.87	10.6223	10.6145	0.0078	0.249	0.309	*DMX	.0002	Light gray to black coating
	Parent	92	17.24	2500	16.22	11.2438	11.2339	0.0099	0.098	0.121	DMY	.0003	Purplish-gray coating
	Welded	92	17.24	2500	15.88	10.9031	10.8951	0.0088	0.081	0.101	DMY	.0003	Purplish-gray coating
	Parent	270	17.24	2500	16.44	11.2743	11.2215	0.0528	0.175	0.217	DMZ	.13	Gray-brown coating
	Welded	270	17.24	2500	15.97	10.8170	10.7851	0.0319	0.109	0.135	DMZ	.13	Gray-brown coating
Steel, C 1010	Parent	30	8.62	1250	14.01	1.3428	1.3428	-0-	-0-	-0-	C7X	.17	Some purple stain
	Welded	30	8.62	1250	13.90	1.7504	1.7500	0.0004	0.014	0.018	C7X	.17	Some purple stain
	Parent	30	13.44	1950	13.99	1.3245	1.3247	(0.0002)	-0-	-0-	D7X	.17	Purple stain
	Welded	30	13.44	1950	14.07	1.7536	1.7537	(0.0001)	-0-	-0-	D7X	.17	Purple stain
	Parent	87	10.34	1500	14.04	1.3304	1.3303	0.0001	0.001	0.001	C7Y	.0002	Purplish-gray stain
	Welded	87	10.34	1500	13.98	1.6643	1.6635	0.0008	0.010	0.012	C7Y	.0002	Purplish-gray stain
	Parent	269	10.34	1500	13.97	1.3277	1.3268	0.0009	0.003	0.004	C7Z	.0002	Deep purple stain
	Welded	269	10.34	1500	14.12	1.7659	1.7622	0.0027	0.010	0.013	C7Z	.0002	Deep purple stain
	Parent	32	17.24	2500	13.99	1.3245	1.3247	(0.0002)	-0-	-0-	*D7X	.0003	No apparent reaction
	Welded	32	17.24	2500	14.07	1.7536	1.7535	0.0001	0.003	0.004	*D7X	.0003	No apparent reaction
	Parent	87	17.24	2500	14.07	1.3272	1.3264	0.0008	0.010	0.012	D7Y	.0002	Purplish-gray stain
	Welded	87	17.24	2500	13.91	1.8419	1.8391	0.0028	0.034	0.042	D7Y	.0002	Purplish-gray stain
	Parent	269	17.24	2500	14.06	1.3557	1.3545	0.0012	0.005	0.006	D7Z	.0002	Purple stain
	Welded	269	17.24	2500	14.11	1.7378	1.7351	0.0027	0.010	0.013	D7Z	.0002	Purple stain
Inconel-718, STA	Parent	30	8.62	1250	14.21	1.4664	1.4662	0.0002	0.007	0.008	CMX	.17	No apparent reaction
	Welded	30	8.62	1250	14.25	2.3084	2.3066	0.0018	0.059	0.073	CMX	.17	No apparent reaction
	Parent	30	13.44	1950	14.19	1.3870	1.3862	0.0008	0.026	0.033	DMX	.17	No apparent reaction
	Welded	30	13.44	1950	14.24	1.9505	1.9450	0.0055	0.180	0.224	DMX	.17	No apparent reaction
	Parent	91	10.34	1500	14.78	3.5443	3.5410	0.0033	0.035	0.043	CMY		Some black stain
	Welded	91	10.34	1500	14.44	3.8809	3.8690	0.0019	0.020	0.025	CMY		Some black stain
	Parent	269	10.34	1500	14.36	1.4512	1.4506	0.0006	0.002	0.003	CMZ	.14	Light brown and purple stain
	Welded	269	10.34	1500	14.28	2.0616	2.0409	0.0207	0.075	0.094	CMZ	.14	Black
	Parent	29	17.24	2500	14.19	1.3870	1.3845	0.0025	0.050	0.106	*DMX	.0002	Light gray to black coating
	Welded	29	17.24	2500	14.24	1.9305	1.9257	0.0048	0.163	0.202	*DMX	.0002	Light gray to black coating
	Parent	92	17.24	2500	14.31	3.3757	3.3714	0.0043	0.046	0.057	DMY	.0003	Light coating, black color
	Welded	92	17.24	2500	14.55	3.7995	3.7861	0.0034	0.036	0.045	DMY	.0003	Light coating, black color
	Parent	270	17.24	2500	14.28	1.4410	1.4400	0.0010	0.004	0.005	DMZ	.13	Dark stain
	Welded	270	17.24	2500	14.42	1.9569	1.9381	0.0188	0.068	0.084	DMZ	.13	Dark stain

TABLE 2.2-4 (cont.)

Material	Specimen Type	Exposure Time, days	Test Pressure MN/m^2	Test Pressure psia	Specimen Surface Area cm^2	Specimen Weights, gm Initial	Specimen Weights, gm Final	Specimen Weights, gm Loss	Penetration Rates pm/sec	Penetration Rates mpy	Test No.	Initial Active Fluoride Content Weight %	Observations
Titanium, 6 Al-4V, STA	Parent	32	8.62	1250	15.13	2.4590	2.4509	0.0081	0.433	0.538	C2X	.0001	Hygroscopic dark brown deposit
	Welded	32	8.62	1250	14.88	2.6547	2.6469	0.0078	0.424	0.527	C2X	.0001	Hygroscopic dark brown deposit
	Parent	32	13.44	1950	15.02	2.3746	2.3633	0.0113	0.606	0.757	D2X	.0001	Hygroscopic yellow-green deposit,
	Welded	32	13.44	1950	14.42	2.0625	2.0506	0.0119	0.668	0.830	D2X	.0001	turning brown after exposure to air.
	Parent	88	10.34	1500	14.71	2.3154	2.3091	0.0063	0.072	0.089	C2Y	.0002	Greenish-gray coating
	Welded	88	10.34	1500	14.71	2.3598	2.3512	0.0086	0.098	0.121	C2Y	.0002	Greenish-gray coating
	Parent	273	10.34	1500	15.07	2.4457	2.4373	0.0084	0.053	0.066	C2Z	.0002	Very hygroscopic yellow-green film,
	Welded	273	10.34	1500	15.03	2.7085	2.6988	0.0097	0.061	0.076	C2Z	.0002	turning brown on hydrolysis.
	Parent	32	17.24	2500	15.02	2.3746	2.3610	0.0136	0.73	0.91	*D2X	.0003	Dull gray coating
	Welded	32	17.24	2500	14.42	2.0625	2.0481	0.0144	0.81	1.01	*D2X	.0003	Dull gray coating
	Parent	88	17.24	2500	14.30	1.8755	1.8689	0.0066	0.077	0.096	D2Y	.0002	Gray coating
	Welded	88	17.24	2500	14.58	2.1412	2.1335	0.0077	0.089	0.111	D2Y	.0002	Gray coating
	Parent	269	17.24	2500	15.14	2.4473	2.4330	0.0143	0.091	0.113	D2Z	.0002	Very hygroscopic yellow-green film,
	Welded	269	17.24	2500	15.03	2.7389	2.7151	0.0238	0.153	0.189	D2Z	.0002	turning brown on hydrolysis.

TABLE 2.2-5

CHEMICAL COMPOSITION OF NITROGEN TRIFLUORIDE RECOVERED FROM STATIC EXPOSURE TESTS WITH METALS

Test No.	Type of Exposure	Composition, Weight Percent							Cylinder of Origin for the NF_3	Table No. in Which Specimen Data are Reported
		NF_3	Active Fluorides as HF	N_2	O_2/CO	CF_4	CO_2	N_2O		
A1X	Liquid/Vapor NF_3, 195 K, 33 days All aluminum alloys	99.19	0.0004	0.24	0.38	0.0083	0	0.18	H81136	2.2-2
A1Y	Liquid/Vapor NF_3, 195 K, 90 days All aluminum alloys	98.93	0.0008	0.52	0.47	0.0072	0	0.075	H81136	2.2-2
A1Z	Liquid/Vapor NF_3, 195 K, 271 days All aluminum alloys	97.98	0.31	0.61	0.53	0.46	0.046	0.068	17319-C	2.2-2
B1X	3.45 MN/m^2 NF_3, 344 K, 32 days All aluminum alloys	99.55	0.051	0.12	0.20	0.0073	0.0053	0.074	H81136	2.2-3
B1Y	3.45 MN/m^2 NF_3, 344 K, 90 days All aluminum alloys	98.73	0.0033	0.77	0.39	0.0070	0.0097	0.083	H81136	2.2-3
B1Z	3.45 MN/m^2 NF_3, 344 K, 271 days All aluminum alloys	98.09	0.38	0.65	0.30	0.45	0.048	0.076	17319-C	2.2-3
C1X	8.62 MN/m^2 NF_3, 344 K, 32 days 2219, T-87 aluminum	99.31	0.053	0.18	0.37	0.0070	0.0046	0.075	H81136	2.2-4
C1Y	10.34 MN/m^2 NF_3, 344 K, 87 days 2219, T-87 aluminum	98.48	0.043	0.24	0.22	0.96	0.012	0.044	H55957	2.2-4
C1Z	10.34 MN/m^2 NF_3, 344 K, 273 days 2219, T-87 aluminum	97.95	0.050	0.64	0.32	0.99	0.0083	0.039	H55957	2.2-4
D1X	13.44 MN/m^2 NF_3, 344 K, 32 days 2219, T-87 aluminum	99.42	0.056	Tr	0.45	0.0078	0.0048	0.058	H81136	2.2-4
*D1X	17.24 MN/m^2 NF_3, 344 K, 32 days 2219, T-87 aluminum	99.30	0.028	0.28	0.33	0.015	0.006	0.033	P178684	2.2-4
D1Y	17.24 MN/m^2 NF_3, 344 K, 87 days 2219, T-87 aluminum	98.62	0.027	0.094	0.20	1.00	0.0079	0.044	H55957	2.2-4
D1Z	17.24 MN/m^2 NF_3, 344 K, 269 days 2219, T-87 aluminum	98.52	0.085	0.17	0.20	0.99	0.0095	0.022	H55957	2.2-4
	Cylinder 17228-C	98.68	0.17	0.20	0.10	0.75	0.013	0.083		
	Cylinder 17319-C	98.72	0.10	0.13	0.45	0.51	0.016	0.070		
	Cylinder H55957	98.68	0.0002	0	0.24	1.03	0	0.048		
	Cylinder H81136	99.56	0.0001	0	0.35	0.009	0	0.074		
	Cylinder P178684	99.68	0.0003	0	0.29	0.017	0	0.014		
A2X	Liquid/Vapor NF_3, 195 K, 33 days Titanium alloys	99.25	Tr.	0.29	0.39	0.0080	0.0024	0.057	H81136	2.2-2

TABLE 2.2-5 (cont.)

Test No.	Type of Exposure	Composition, Weight Percent: NF_3	Active Fluorides as HF	N_2	O_2/CO	CF_4	CO_2	N_2O	Cylinder of Origin for the NF_3	Table No. in Which Specimen Data are Reported
A2Y	Liquid/Vapor NF_3, 195 K, 90 days Titanium alloys	99.57	0.0001	Tr.	0.36	0.0072	0	0.060	H81136	2.2-2
A2Z	Liquid/Vapor NF_3, 195 K, 271 days Titanium alloys	98.42	0.10	0.48	0.47	0.45	0.017	0.054	17319-C	2.2-2
B2X	3.45 MN/m^2 NF_3, 344 K, 32 days Titanium alloys	99.43	0.0006	0.16	0.21	0.0074	0.0040	0.19	H81136	2.2-3
B2Y	3.45 MN/m^2 NF_3, 344 K, 90 days Titanium alloys	98.88	0.0021	0.65	0.38	0.0071	0.011	0.073	H81136	2.2-3
B2Z	3.45 MN/m^2 NF_3, 344 K, 271 days Titanium alloys	98.44	0.0049	0.72	0.24	0.49	0.024	0.089	17319-C	2.2-3
C2X	8.62 MN/m^2 NF_3, 344 K, 32 days Titanium 6Al-4V	98.25	0.0006	1.08	0.59	0.0074	0.0024	0.078	H81136	2.2-4
C2Y	10.34 MN/m^2 NF_3, 344 K, 88 days Titanium 6Al-4V	98.45	0.028	0.21	0.28	0.98	0.0047	0.040	H55957	2.2-4
C2Z	10.34 MN/m^2 NF_3, 344 K, 273 days Titanium 6Al-4V	97.86	0.037	0.73	0.36	0.97	0.0089	0.037	H55957	2.2-4
D2X	13.44 MN/m^2 NF_3, 344 K, 32 days Titanium 6Al-4V	No Data							H81136	2.2-4
*D2X	17.24 MN/m^2 NF_3, 344 K, 32 days Titanium 6Al-4V	99.57	0.006	0.090	0.29	0.015	0.006	0.027	P178684	2.2-4
D2Y	17.24 MN/m^2, 344 K, 88 days Titanium 6Al-4V	98.52	0.014	0.17	0.25	0.99	0.0053	0.048	H55957	2.2-4
D2Z	17.24 MN/m^2 NF_3, 344 K, 269 days Titanium 6Al-4V	98.58	0.020	0.15	0.22	1.00	0.011	0.026	H55957	2.2-4
A3X	Liquid/Vapor NF_3, 195 K, 33 days Aluminum-Bronze 623	99.48	0.0002	Tr.	0.41	0.0080	0.0024	0.099	H81136	2.2-2
A3Y	Liquid/Vapor NF_3, 195 K, 90 days Aluminum-Bronze 623	99.18	0.002	0.34	0.41	0.0073	0	0.058	H81136	2.2-2
A3Z	Liquid/Vapor NF_3, 195 K, 270 days Aluminum-Bronze 623	98.47	0.10	0.33	0.57	0.45	0.017	0.058	17319-C	2.2-2
B3X	3.45 MN/m^2 NF_3, 344 K, 32 days Aluminum-Bronze 623	99.18	0.0052	0.40	0.29	0.0077	0.0053	0.11	H81136	2.2-3
B3Y	3.45 MN/m^2 NF_3, 344 K, 90 days Aluminum-Bronze 623	98.48	0.0084	1.11	0.24	0.0077	0.015	0.14	H81136	2.2-3

TABLE 2.2-5 (cont.)

Test No.	Type of Exposure	Composition, Weight Percent: NF_3	Active Fluorides as HF	N_2	O_2/CO	CF_4	CO_2	N_2O	Cylinder of Origin for the NF_3	Table No. in Which Specimen Data are Reported
B6X	3.45 MN/m^2 NF_3, 344 K, 32 days 17-4 PH SS, H-1025	99.47	0.0002	0.19	0.22	0.0078	0.0055	0.11	H81136	2.2-3
B6Y	3.45 MN/m^2 NF_3, 344 K, 90 days 17-4 PH SS, H-1025	98.77	<.0002	0.71	0.43	0.0066	0.0070	0.065	H81136	2.2-3
B6Z	3.45 MN/m^2 NF_3, 344 K, 271 days 17-4 PH SS, H-1025	96.62	0.052	2.18	0.59	0.45	0.030	0.080	17319-C	2.2-3
B7X	3.45 MN/m^2 NF_3, 344 K, 32 days 1010 Steel	99.54	0.0051	0.13	0.21	0.0077	0.0052	0.10	H81136	2.2-3
B7Y	3.45 MN/m^2 NF_3, 344 K, 90 days 1010 Steel	99.14	<.0002	0.43	0.34	0.0068	0.0084	0.064	H81136	2.2-3
B7Z	3.45 MN/m^2 NF_3, 344 K, 271 days 1010 Steel	98.55	0.11	0.80	Tr.	0.46	0.020	0.070	17319-C	2.2-3
C7X	8.62 MN/m^2 NF_3, 344 K, 30 days 1010 Steel	97.28	0.45	1.47	Tr.	0.66	0.078	0.057	17228-C	2.2-4
C7Y	10.34 MN/m^2 NF_3, 344 K, 87 days 1010 Steel	98.23	0.071	0.35	0.32	0.96	0.018	0.046	H55957	2.2-4
C7Z	10.34 MN/m^2 NF_3, 344 K, 269 days 1010 Steel	97.83	0.068	0.76	0.33	0.96	0.011	0.029	H55957	2.2-4
D7X	13.44 MN/m^2 NF_3, 344 K, 30 days 1010 Steel	97.94	0.44	0.50	0.25	0.65	0.16	0.065	17228-C	2.2-4
*D7X	17.24 MN/m^2 NF_3, 344 K, 32 days 1010 Steel	99.66	0.0004	0.11	0.18	0.014	0.005	0.033	P178684	2.2-4
D7Y	17.24 MN/m^2 NF_3, 344 K, 87 days 1010 Steel	98.43	0.032	0.24	0.24	1.00	0.014	0.045	H55957	2.2-4
D7Z	17.24 MN/m^2 NF_3, 344 K, 269 days 1010 Steel	98.47	0.078	0.21	0.21	0.99	0.013	0.022	H55957	2.2-4
A20Y	Liquid/Vapor NF_3, 195 K, 91 days Carpenter Custom 455	99.26	0.025	0.30	0.36	0.014	0	0.034	P178684	2.2-2
B20Y	3.45 MN/m^2 NF_3, 344 K, 91 days Carpenter Custom 455	99.44	0.0005	0.28	0.24	0.014	0.0045	0.022	P178684	2.2-3
AMX	Liquid/Vapor NF_3, 195 K, 34 days All other metal alloys	98.69	0.086	0.14	0.39	0.59	0.028	0.073	17319-C	2.2-2

TABLE 2.2-5 (cont.)

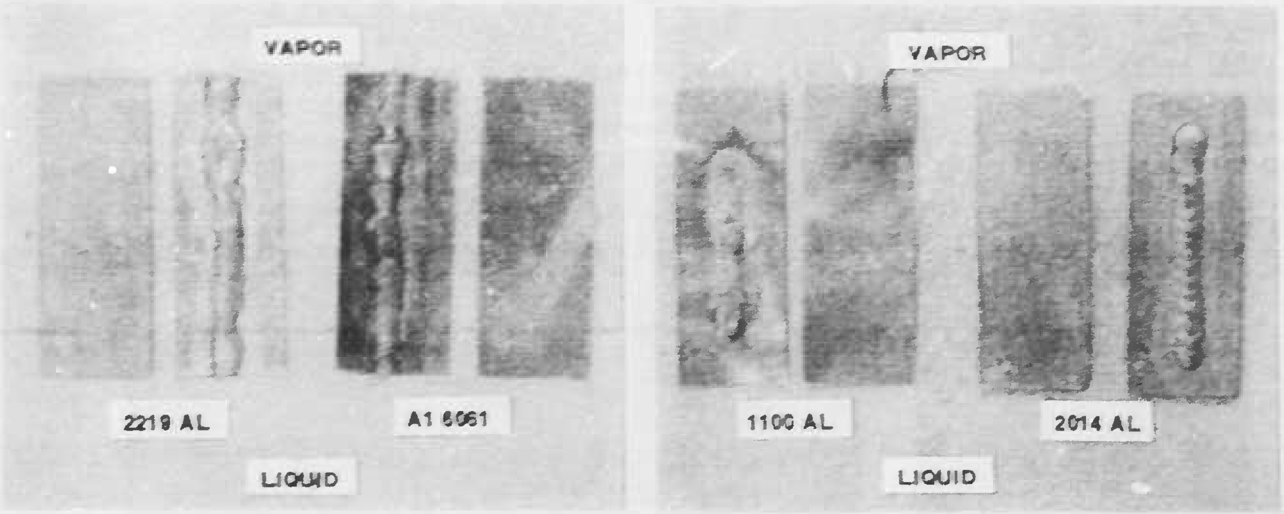
Test No.	Type of Exposure	Composition, Weight Percent: NF_3	Active Fluorides as HF	N_2	O_2/CO	CF_4	CO_2	N_2O	Cylinder of Origin for the NF_3	Table No. in Which Specimen Data are Reported
B3Z	3.45 MN/m^2 NF_3, 344 K, 270 days Aluminum-Bronze 623	98.14	0.041	0.94	0.29	0.45	0.055	0.076	17319-C	2.2-3
A4X	Liquid/Vapor NF_3, 195 K, 33 days Tungsten, 2% Throiam	99.28	0.0003	0.26	0.39	0.0078	0.0085	0.062	H81136	2.2-2
A4Y	Liquid/Vapor NF_3, 195 K, 90 days Tungsten, 2% Thoriam	99.48	0.0001	Tr.	0.44	0.0073	0	0.071	H81136	2.2-2
A4Z	Liquid/Vapor NF_3, 195 K, 270 days Tunbsten, 2% Thoriam	98.67	0.10	0.36	0.34	0.43	0.032	0.058	17319-C	2.2-2
B4X	3.45 MN/m^2 NF_3, 344 K, 33 days Tungsten, 2% Thoriam	99.21	0.0042	0.39	0.30	0.0075	0.015	0.075	H81136	2.2-3
B4Y	3.45 MN/m^2 NF_3, 344 K, 90 days Tungsten, 2% Thoriam	96.99	0.0026	0.56	0.36	0.0072	0.012	0.073	H81136	2.2-3
B4Z	3.45 MN/m^2 NF_3, 344 K, 270 days Tungsten, 2% Thoriam	97.88	0.054	1.49	Tr.	0.47	0.019	0.083	17319-C	2.2-3
A5X	Liquid/Vapor NF_3, 195 K, 33 days Beryllium Copper	99.48	0.0004	0.055	0.39	0.0077	0.0026	0.056	H81136	2.2-2
A5Y	Liquid/Vapor NF_3, 195 K, 90 days Beryllium Copper	99.43	0.0076	0.12	0.38	0.0072	0	0.056	H81136	2.2-2
A5Z	Liquid/Vapor NF_3, 195 K, 269 days Beryllium Copper	98.39	0.073	0.50	0.45	0.48	0.040	0.071	17319-C	2.2-2
B5X	3.45 MN/m^2 NF_3, 344 K, 33 days Beryllium Copper	98.99	0.0025	0.57	0.35	0.0073	0.011	0.067	H81136	2.2-3
B5Y	3.45 MN/m^2 NF_3, 344 K, 90 days Beryllium Copper	98.66	<.0002	0.31	0.46	0.0068	0.0068	0.055	H81136	2.2-3
B5Z	3.45 MN/m^2 NF_3, 344 K, 270 days Beryllium Copper	98.31	0.12	0.75	0.20	0.48	0.051	0.090	17319-C	2.2-3
A6X	Liquid/Vapor NF_3, 195 K, 33 days 17-4 PH SS, H-1025	99.64	Tr.	0.096	0.36	0.0078	0	0.10	H81136	2.2-2
A6Y	Liquid/Vapor NF_3, 195 K, 90 days 17-4 PH SS, H-1025	99.20	<.0001	0.32	0.42	0.0072	0	0.056	H81136	2.2-2
A6Z	Liquid/Vapor NF_3, 195 K, 269 days 17-4 PH SS, H-1025	97.11	0.098	1.40	0.84	0.45	0.033	0.065	17319-C	2.2-2

TABLE 2.2-5 (cont.)

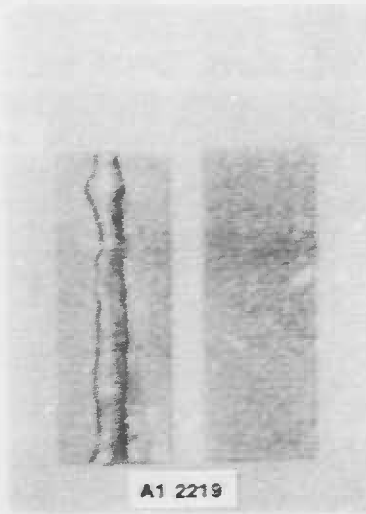
Test No.	Type of Exposure	Composition, Weight Percent: NF_3	Active Fluorides as HF	N_2	O_2/CO	CF_4	CO_2	N_2O	Cylinder of Origin for the NF_3	Table No. in Which Specimen Data are Reported
AMY	Liquid/Vapor NF3, 195 K, 91 days All other metal alloys	99.75	0.0002	0	0.19	0.017	0	0.044	P178684	2.2-2
AMZ	Liquid/Vapor NF3, 195 K, 274 days All other metal alloys	98.69	0.18	0.19	0.37	0.46	0.029	0.077	17319-C	2.2-2
BMX	3.45 MN/m^2 NF3, 344 K, 30 days All other metal alloys	98.03	0.47	0.58	0.31	0.48	0.075	0.064	17319-C	2.2-3
BMY	3.45 MN/m^2 NF3, 344 K, 91 days All other metal alloys	99.56	0.022	Tr.	0.32	0.015	0.020	0.060	P178684	2.2-[illegible]
BMZ	3.45 MN/m^2 NF3, 344 K, 270 days All other metal alloys	98.62	0.27	0.54	0.15	0.46	0.033	0.11	17319-C	2.2-3
CMX	8.62 MN/m^2 NF3, 344 K, 30 days 301 SS, VM-200+250, Inconel -718	98.05	0.56	0.48	0.14	0.65	0.043	0.081	17228-C	2.2-4
CMY	10.34 MN/m^2 NF3, 344 K,91 days 301 SS, VM-200+250, Inconel -718	99.62	0.019	Tr.	0.32	0.014	0.0023	0.022	P178684	2.2-4
CMZ	10.34 MN/m^2 NF3, 344 K, 269 days 301 SS, VM-200+250, Inconel -718	98.24	0.46	0.49	Tr.	0.68	0.054	0.078	84% 17228-C 16% H55957	2.2-4
DMX	13.44 MN/m^2 NF3, 344 K, 30 days 301 SS, VM-200+250, Inconel -718	97.96	0.60	0.55	0.12	0.65	0.039	0.082	17228-C	2.2-4
DMY	17.24 MN/m^2 NF3, 344 K, 92 days 301 SS, VM-200 +250, Inconel -718	99.59	0.029	Tr.	0.32	0.015	0.0064	0.031	P178684	2.2-4
*DMX	17.24 MN/m^2 NF3, 344 K, 29 days 301 SS, VM-200+250, Inconel -718	99.15	0.028	Tr.	0.31	0.45	0.012	0.052	H55957 P178684	2.2-4
DMZ	17.24 MN/m^2 NF3, 344 K, 270 days 301 SS, VM-200+250, Inconel -718	98.32	0.42	0.43	Tr.	0.72	0.035	0.077	74% 17228-C 26% H55957	2.2-4

2.2, Static Exposure Tests (cont.)

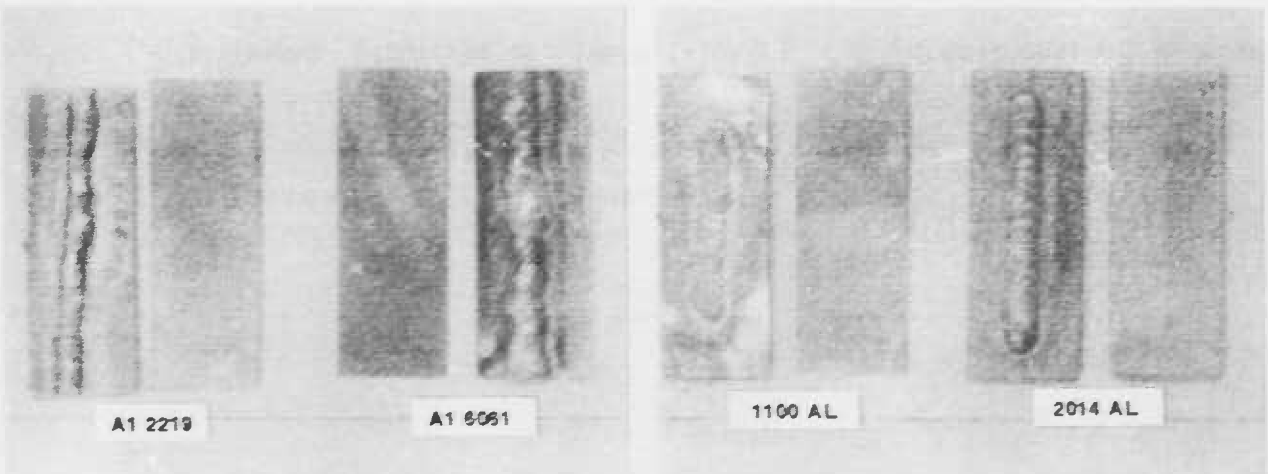
The metal coupons which were subjected to the 270 day exposure tests were photographed in the condition in which they were removed from the test containers and the photographs are presented in Figures 2.2.1 through 2.2.11.



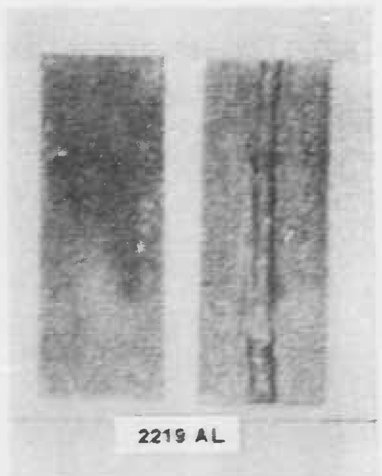
Conditions: Liquid/Vapor, 195°K (-108°F)



Conditions:
10.34 MN/m^2 (1500 psia),
344°K (160°F)

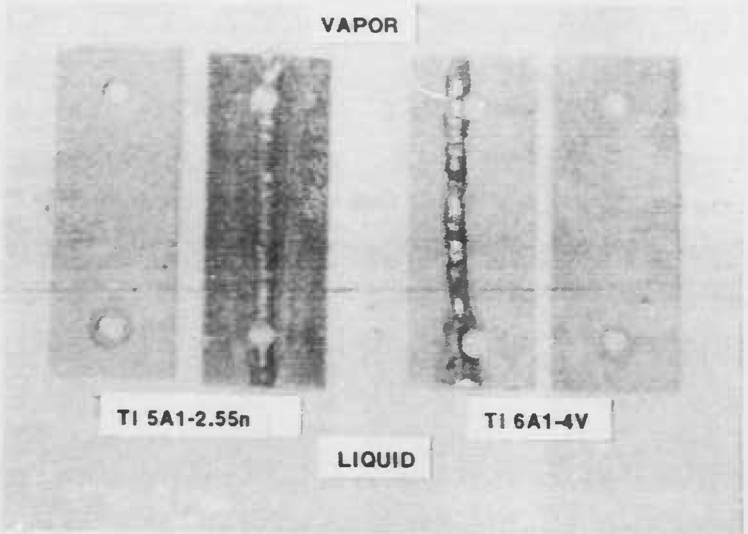


Conditions: 3.45 MN/m^2 (500 psia), 344°K (160°F)

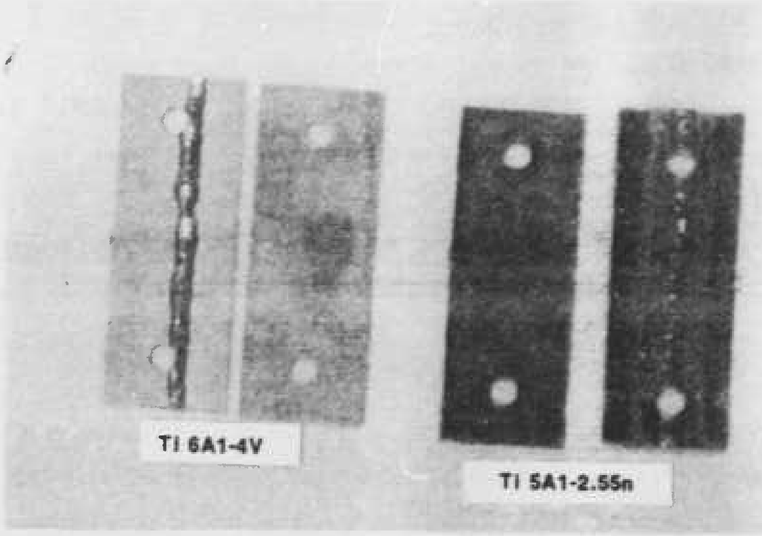


Conditions:
17.24 MN/m^2 (2500 psia),
344°K (160°F)

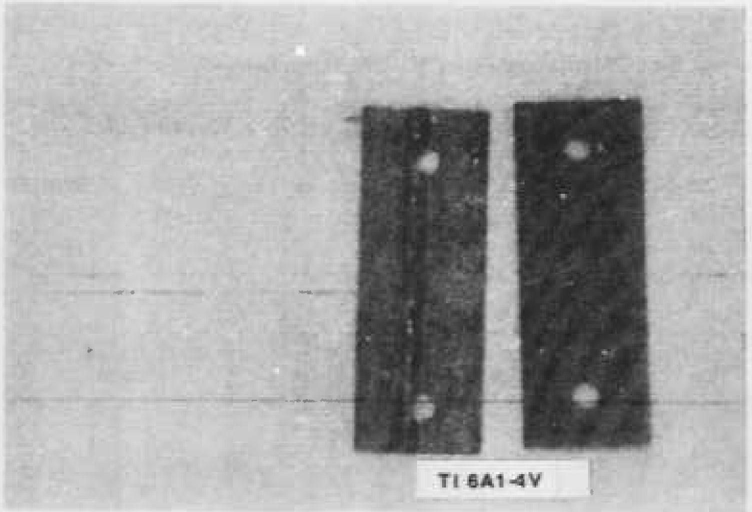
Figure 2.2.1. Aluminum Specimens After 9 Months Static Exposure to Nitrogen Trifluoride



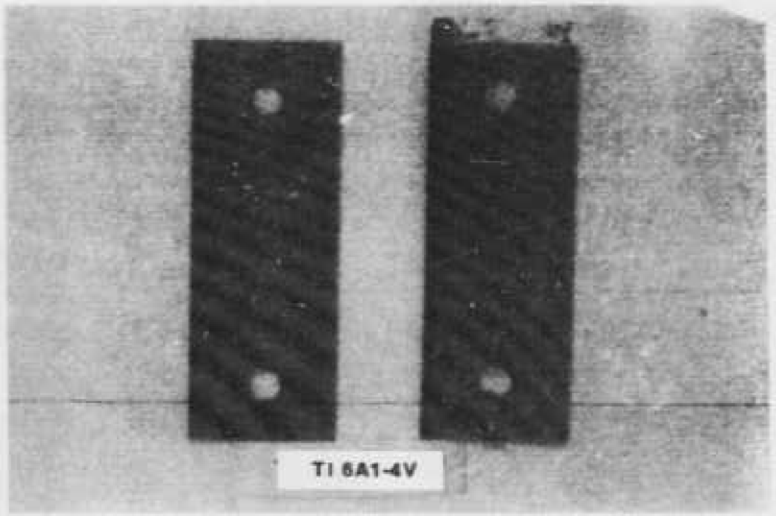
Conditions: Liquid/Vapor, 195°K (-108°F)



Conditions: 3.45 Mn/m^2 (500 psia), 344°K (160°F)

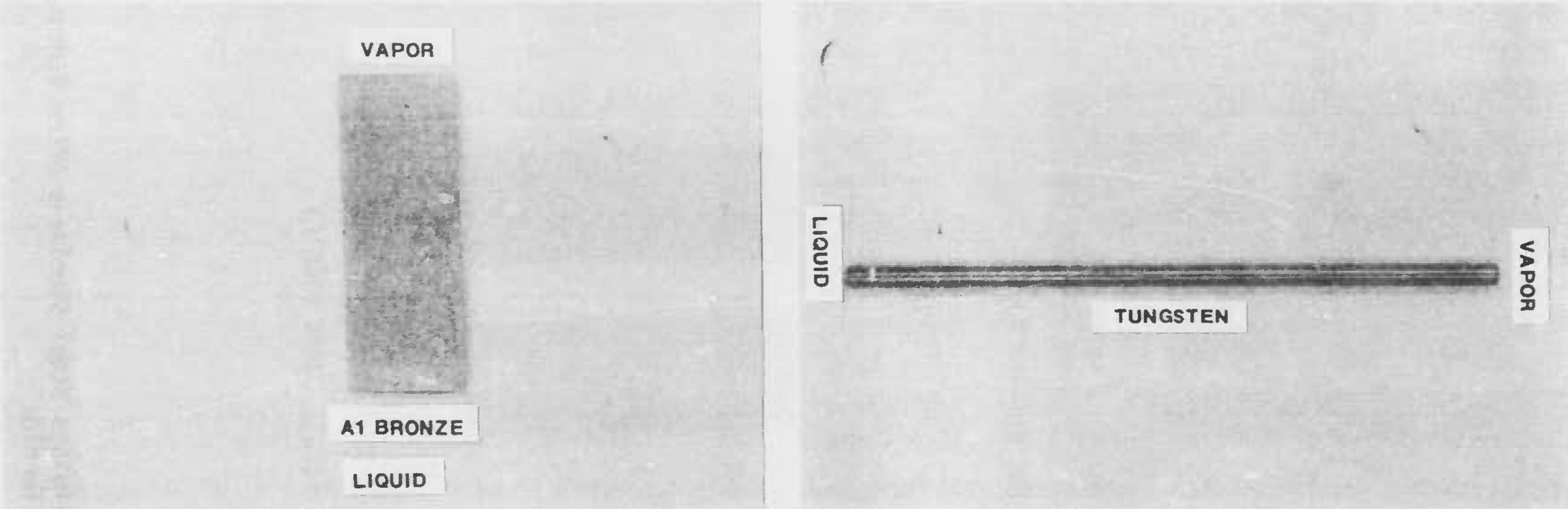


Conditions: 10.34 MN/m^2 (1500 psia), 344°K (160°F)

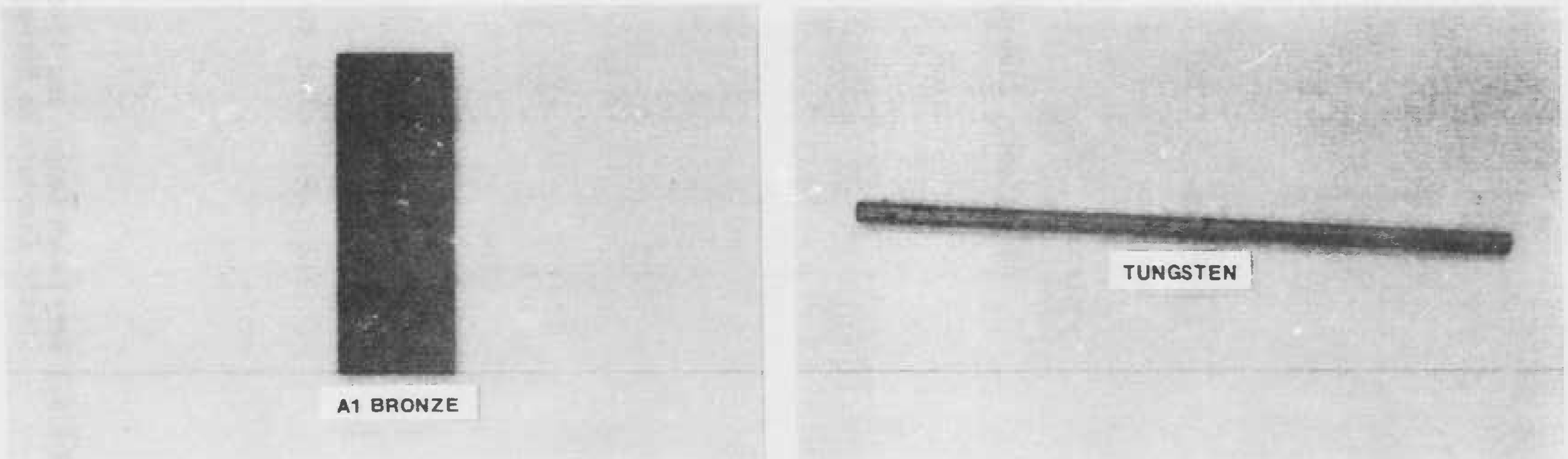


Conditions: 17.24 MN/m^2 (2500 psia), 344°K (160°F)

Figure 2.2.2. Titanium Specimens After 9 Months Static Exposure to Nitrogen Trifluoride

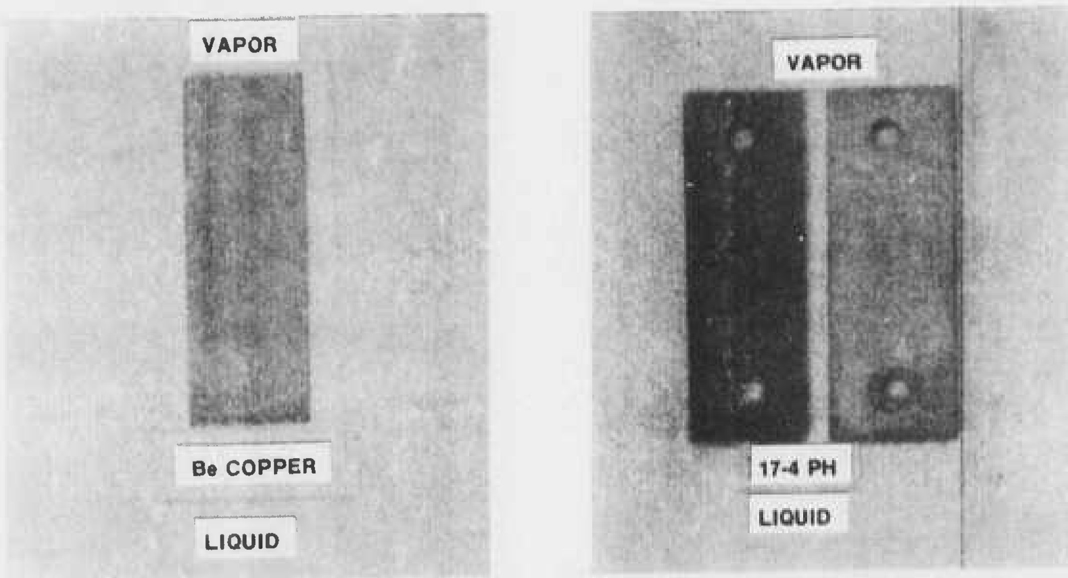


Conditions: Liquid/Vapor, 195°K (-108°F)

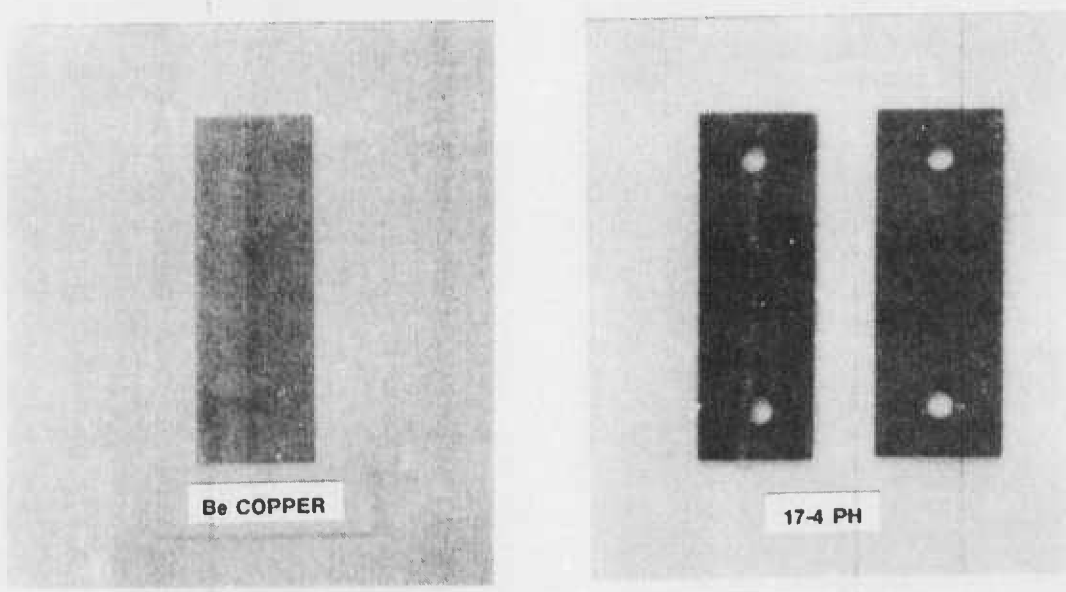


Conditions: 3.45 MN/m^2 (500 psia), 344°K (160°F)

Figure 2.2.3. Aluminum Bronze and Tungsten Specimens After 9 Months Static Exposure to Nitrogen Trifluoride

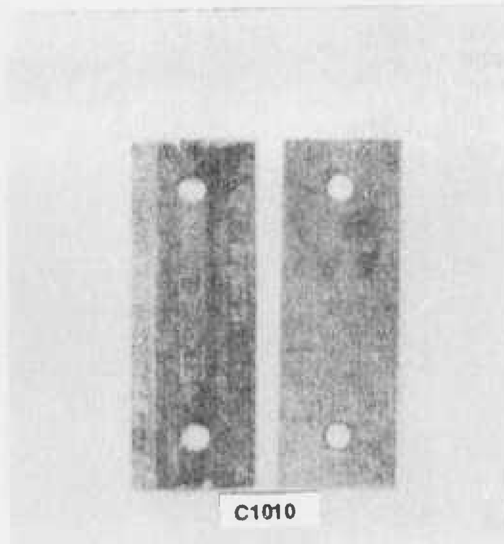


Conditions: Liquid/Vapor, 195°K (-108°F)

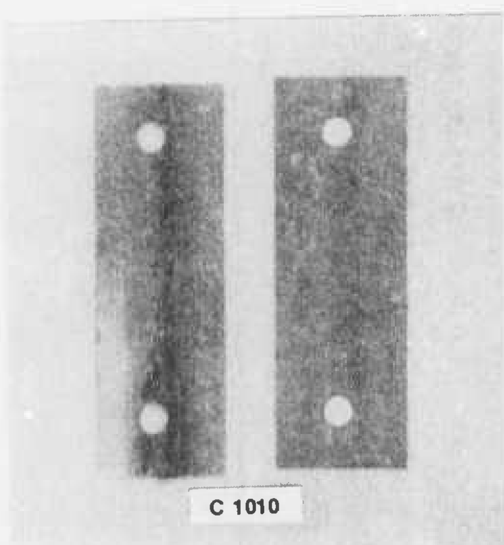


Conditions: 3.45 MN/m^2 (500 psia), 344°K (160°F)

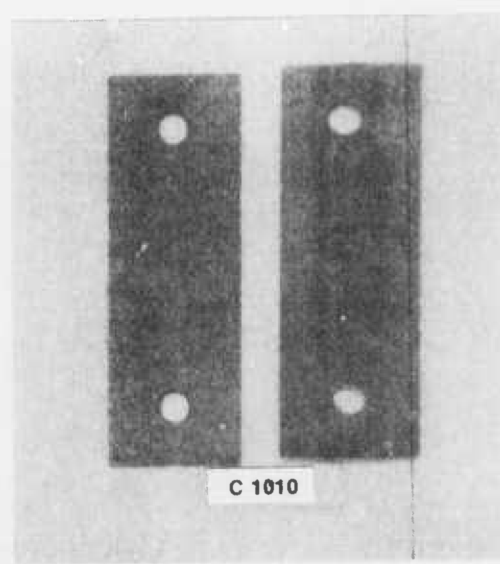
Figure 2.2.4. Beryllium Copper and 17-4 PH Stainless Steel Specimens After 9 Months Static Exposure to Nitrogen Trifluoride



Conditions: 3.45 MN/m^2 (500 psia), 344°K (160°F)

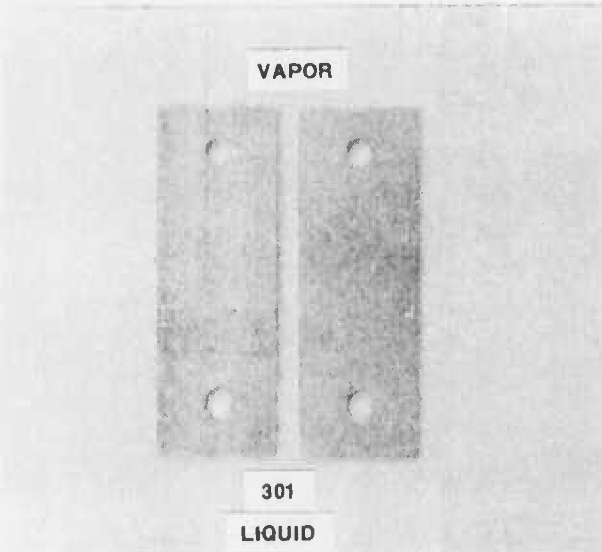


Conditions: 10.34 MN/m^2 (1500 psia), 344°K (160°F)

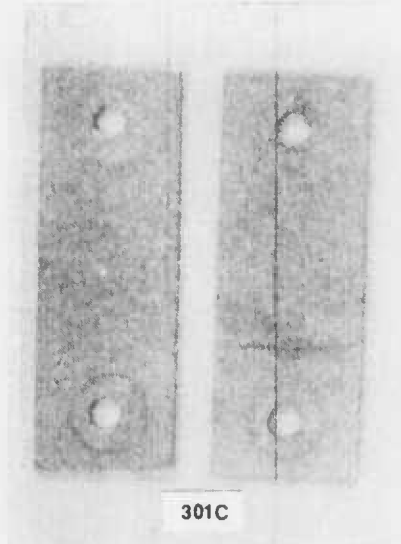


Conditions: 17.24 MN/m^2 (2500 psia), 344°K (160°F)

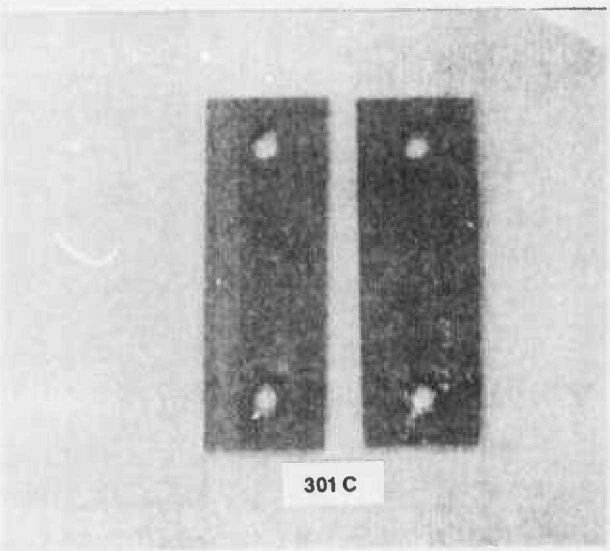
Figure 2.2.5. C1010 Steel Specimens After 9 Months Static Exposure to Nitrogen Trifluoride



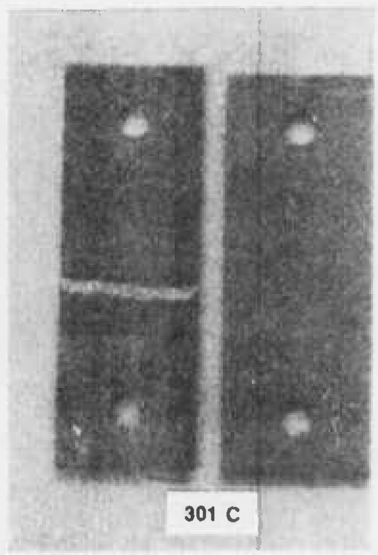
Conditions: Liquid/Vapor, 195°K (-108°F)



Conditions:
10.34 MN/m^2 (1500 psia),
344°K (160°F)

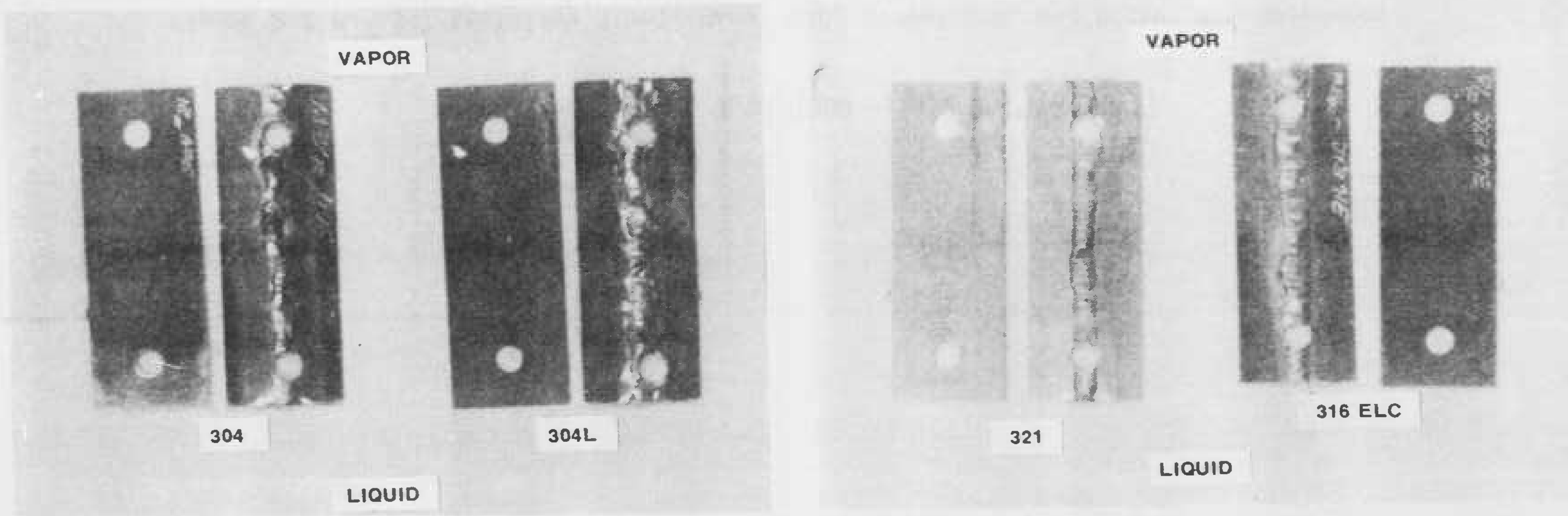


Conditions: 3.45 MN/m^2 (500 psia),
344°K (160°F)

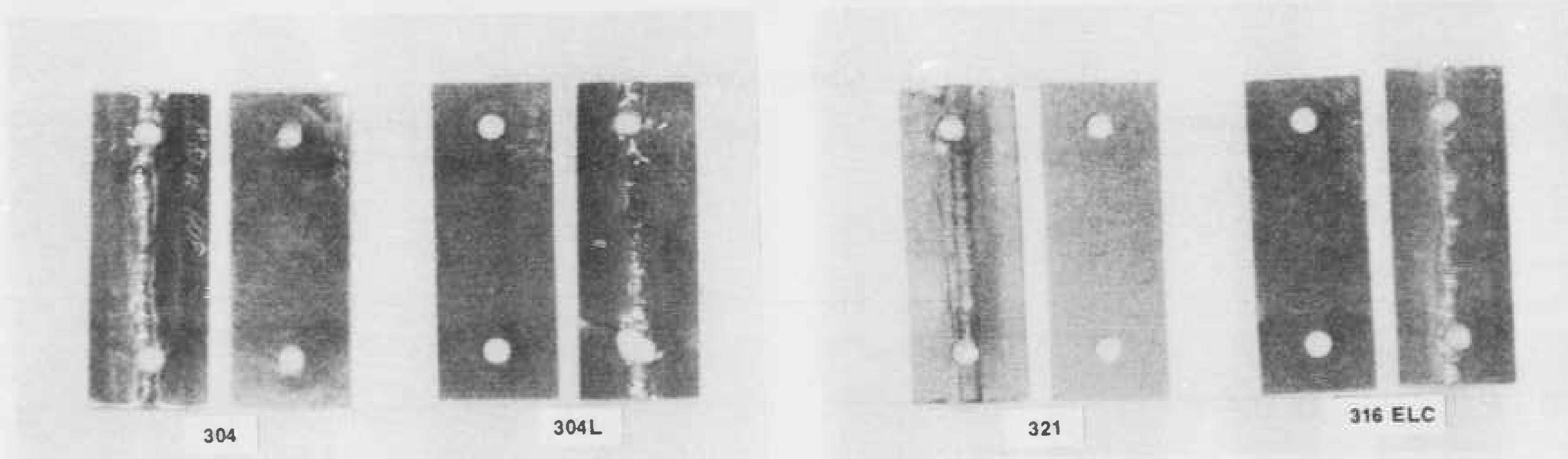


Conditions:
17.24 MN/m^2 (2500 psia),
344°K (160°F)

Figure 2.2.6. 301 Cryoformed Stainless Steel Specimens After 9 Months Exposure to Nitrogen Trifluoride

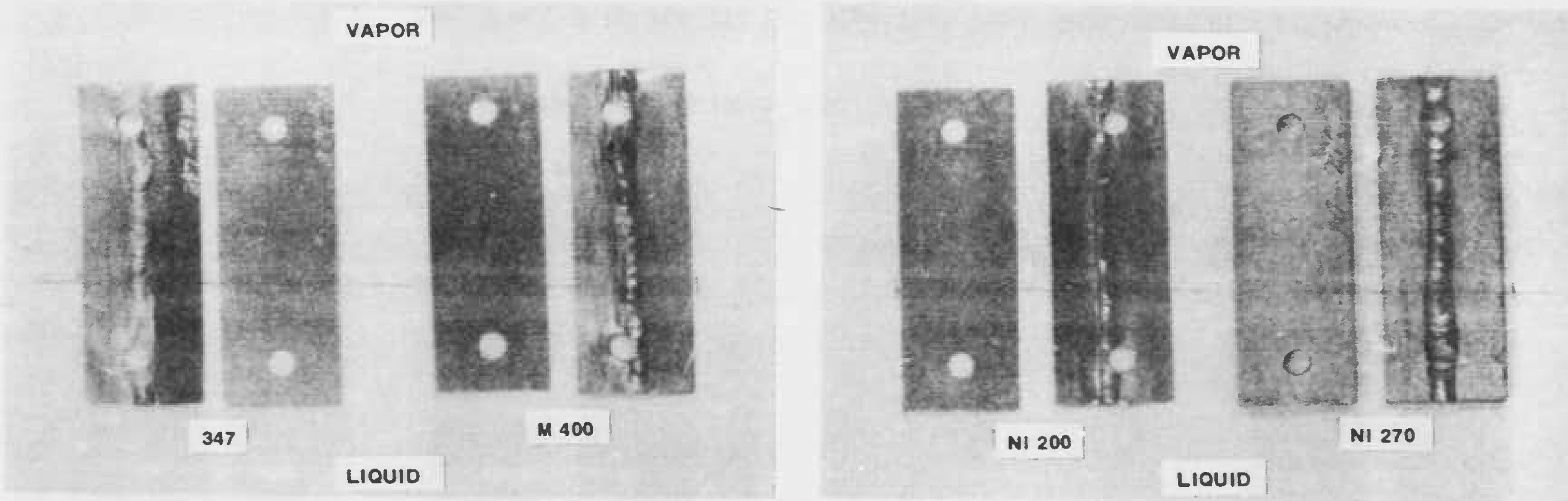


Conditions: Liquid/Vapor, 195°K (-108°F)

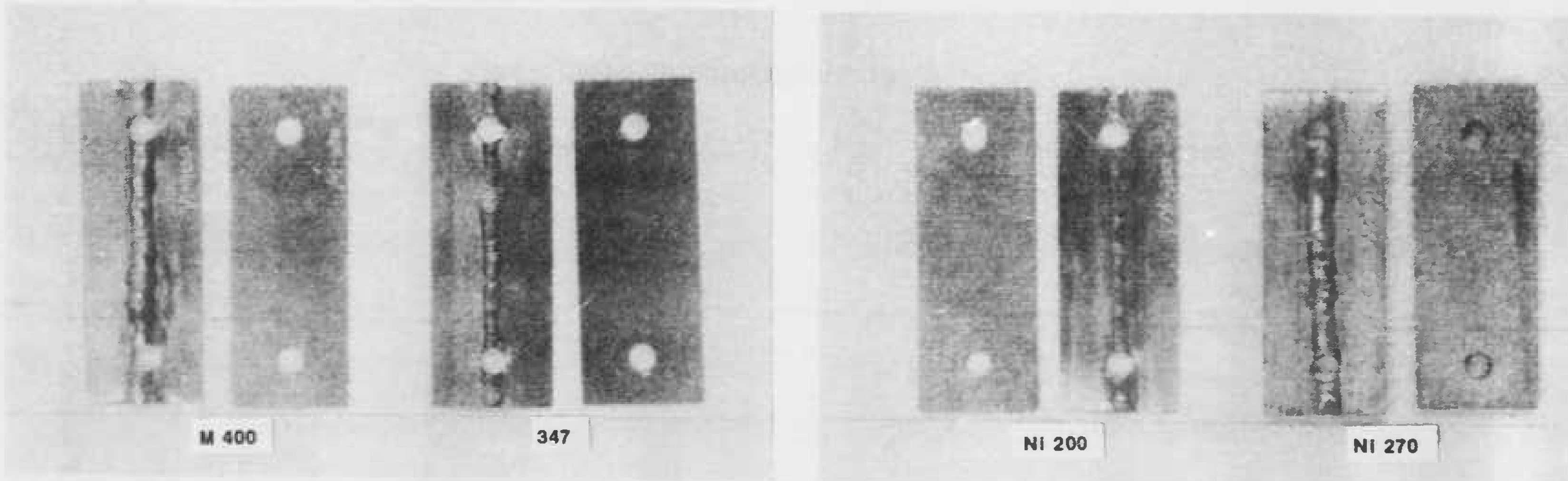


Conditions: 3.45 MN/m^2 (500 psia), 344°K (160°F)

Figure 2.2.7. 304, 304L, 321, and 316 ELC Stainless Steel Specimens After 9 Months Static Exposure to Nitrogen Trifluoride

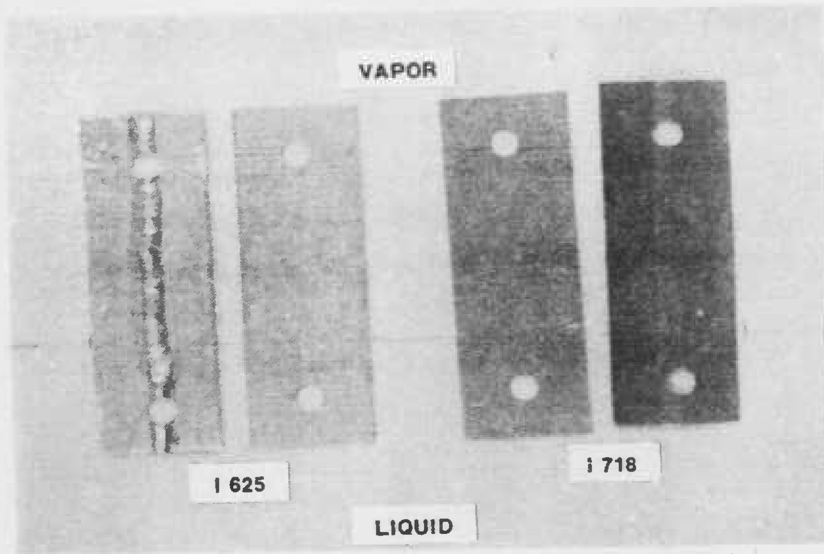


Conditions: Liquid/Vapor, 195°K (-108°F)

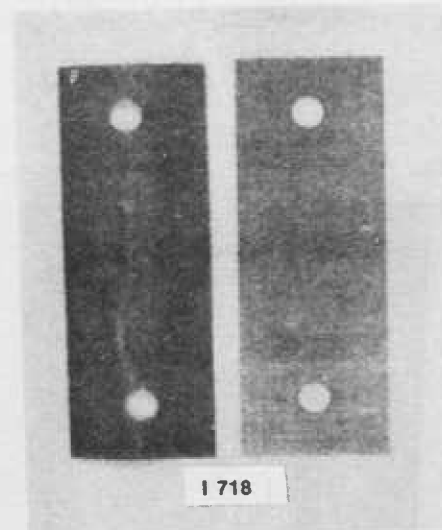


Conditions: 3.45 MN/m^2 (500 psia), 344°K (160°F)

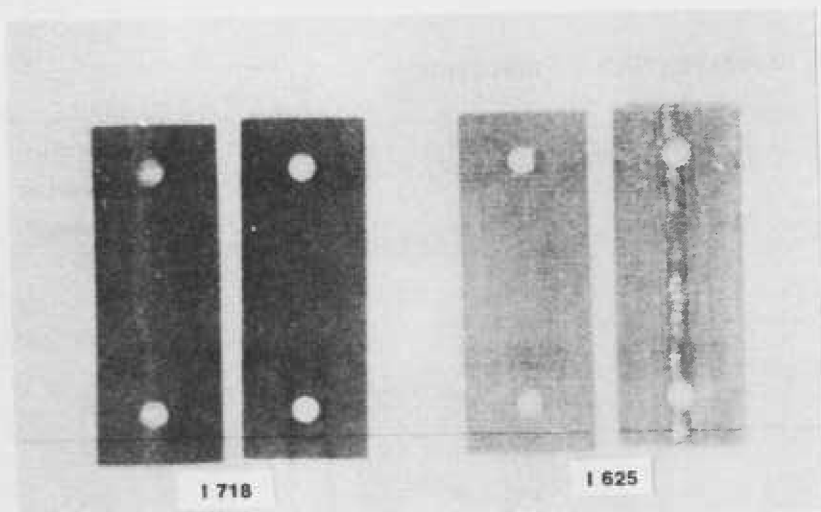
Figure 2.2.8. 347 Stainless Steel, Monel 400, Nickel 200, and Nickel 270 Specimens After 9 Months Static Exposure to Nitrogen Trifluoride



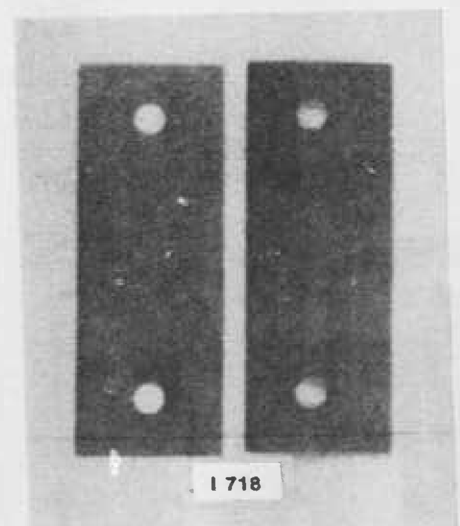
Conditions: Liquid/Vapor, 195°K (-108°F)



Conditions: 10.34 MN/m^2 (1500 psia), 344°K (160°F)

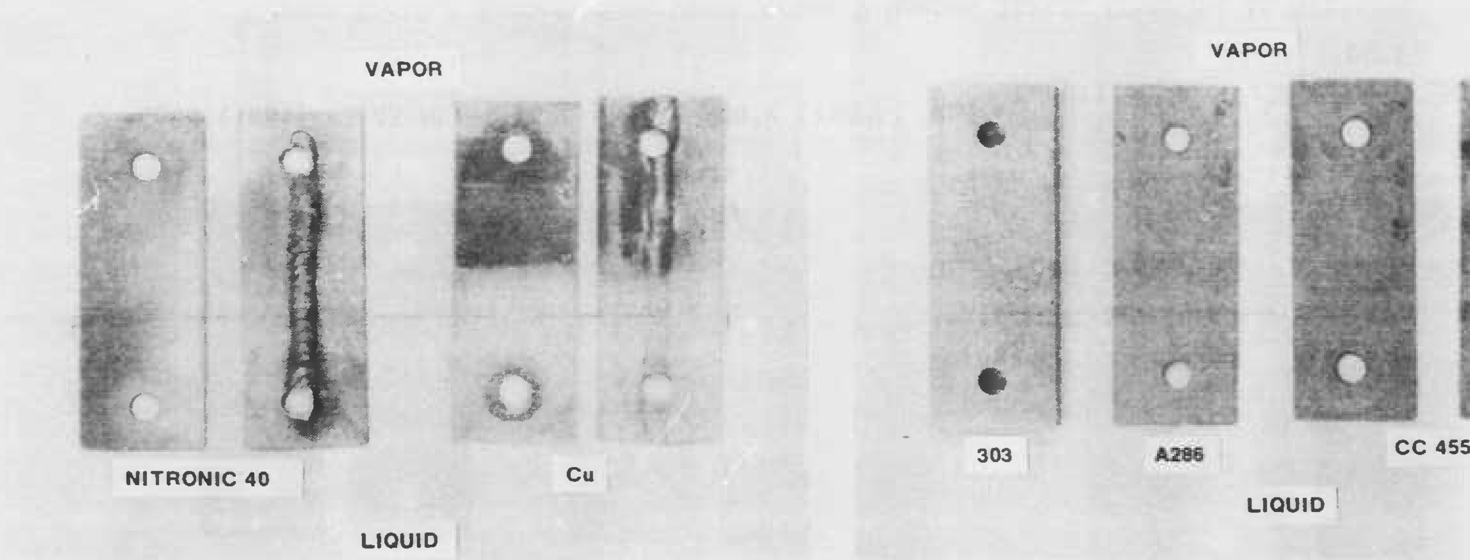


Conditions: 3.45 MN/m^2 (500 psia), 344°K (160°F)

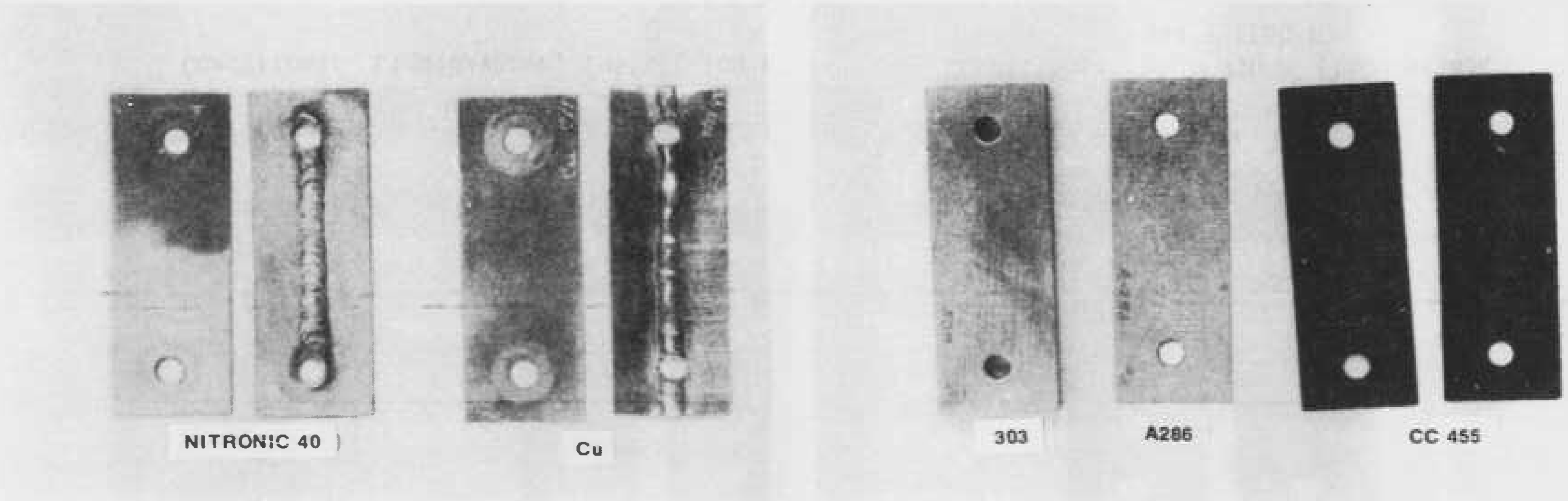


Conditions: 17.24 MN/m^2 (2500 psia), 344°K (160°F)

Figure 2.2.9. Inconel Specimens After 9 Months Static Exposure to Nitrogen Trifluoride

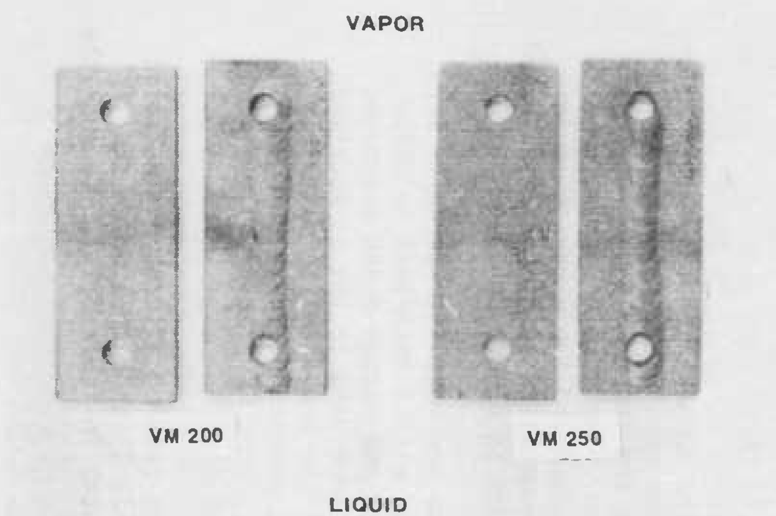


Conditions: Liquid/Vapor, 195 °K (-108°F)

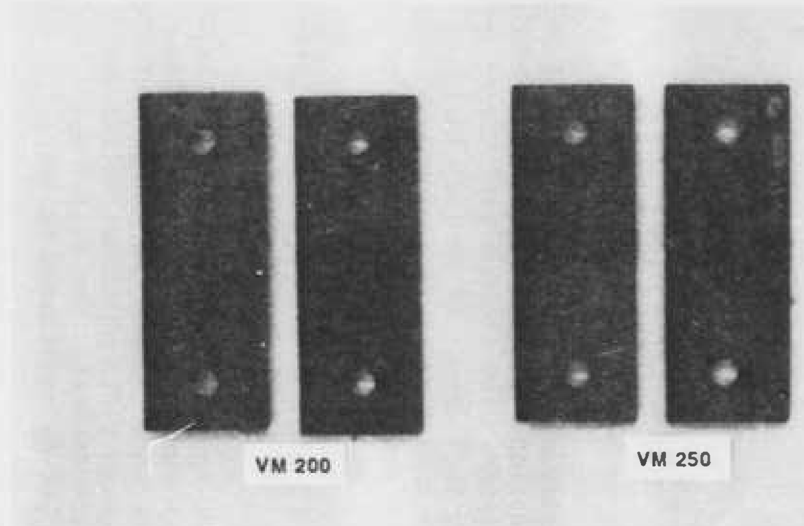


Conditions: 3.45 M/m^2 (500 psia), 344°K (160°F)

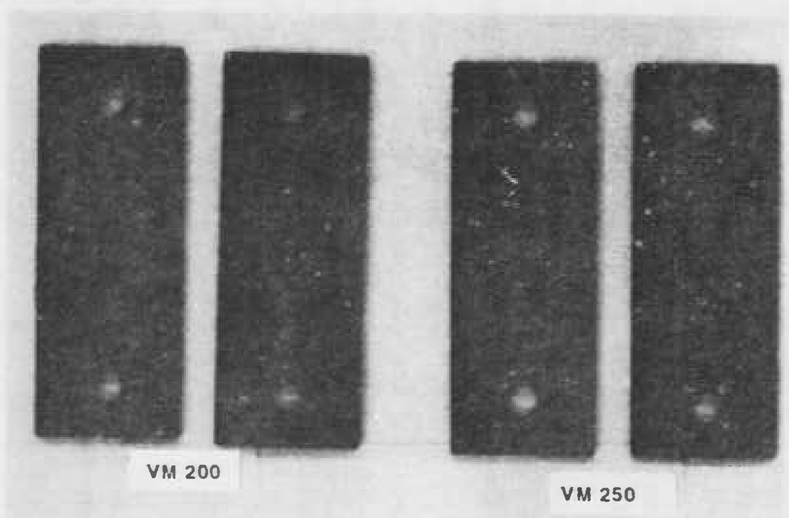
Figure 2.2.10. Nitronic 40, Copper OFHC, 303 Stainless Steel, A286, and Carpenter Custom 455 Specimens After 9 Months Static Exposure to Nitrogen Trifluoride



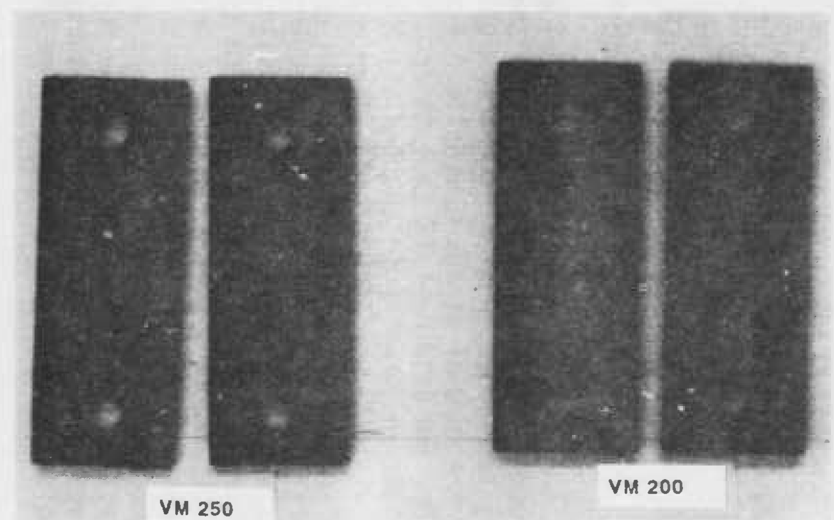
Conditions: Liquid/Vapor, 195°F (-108°F)



Conditions: 3.45 MN/m^2 (500 psia), 344°K (160°F)



Conditions: 10.34 MN/m^2 (1500 psia), 344°K (160°F)



Conditions: 17.24 MN/m^2 (2500 psia), 344°K (160°F)

Figure 2.2.11. Maraging Steel Specimens After 9 Months Static Exposure to Nitrogen Trifluoride

2.2, Static Tests (cont.)

2.2.2 Static Exposure Tests with Non-Metallic Materials

2.2.2.1 Apparatus and Procedures

The non-metal candidates used in the tests can be divided into five categories, (1) thermoplastics, (2) elastomers, (3) graphites, (4) lubricants, and (5) a thermosetting polymer. The thermoplastics were tested in the form of strips nominally 6.4 cm (2.5 in.) long, .64 cm (.25 in.) wide, and 0.33 cm (.13 in.) thick. The thermoplastics were evaluated by weight and appearance changes which occurred and by changes in the modulus of rigidity per ASTM D 1043. The elastomers were tested in the form of "O"-rings; and evaluated by weight and appearance changes, and physical testing for tensile strength, modulus at 100% elongation, ultimate elongation and Wallace hardness per ASTM D 1414. The graphites were in the form of plates 2.5 cm (1 in.) long, 1.2 cm (0.5 in.) wide, and 0.25 cm (0.1 in.) thick; and they were evaluated by weight and appearance changes. The lubricants were tested by placing samples in aluminum cups for physical confinement, and were evaluated by weight and appearance changes. The thermosetting plastic, Kevlar was tested in the form of woven cloth which was rolled and placed in 304 L stainless steel tubes for confinement. The Kevlar was evaluated by weight and appearance changes and by resistance to tear.

The non-metal specimens were exposed for nominal periods of 30, 90 and 270 days to nitrogen trifluoride in liquid- and vapor-phases at 195 K (-78 C) and at 344 K and 3.45 MN/m^2 (500 psia) for all candidates and with selected candidates at pressures up to 17.24 MN/m^2 (2500 psig). The test matrix is shown in Table 2.2-6. The number 2 in the matrix refers to duplicate samples being tested at the same condition; the number 4 in the matrix indicates that one set of duplicates are in the liquid phase and one set of duplicates are in the vapor phase. The grease samples were evaluated as single samples in the liquid phase and the vapor phase in the 195 K temperature environment. The non-metallic specimens were tested in the 304 L stainless steel containers shown in Figure 2.1.3.

Prior to exposure, the non-metallic specimens were, except for the carbon and the grease, washed with a detergent solution (Turco Plaudit), rinsed with deionized water and vacuum dried overnight at 333 K (140 F). The carbons and greases were used in the as-received condition. The compositional definition of the non-metallic materials is presented in Appendix A for the reader's convenience.

2.2.2.2 Experimental Results

The data obtained from testing all the non-metallic materials except the elastomers at 195 K (-78 C) in both liquid- and vapor-phase nitrogen trifluoride are presented in Table 2.2-7. Besides the

TABLE 2.2-6

STATIC COMPATIBILITY TEST MATRIX FOR NON-METALS

Material	Vapor/Liquid NF_3 Exposure -78.5°C, ~200 psia			Gaseous NF_3 Exposures 160°F, 500 psia			160°F, 1500 psia			160°F, 2500 psia		
	30 Days	90 Days	270 Days	30 Days	90 Days	270 Days	30 Days	90 Days	270 Days	30 Days	90 Days	270 Days
Polytetrafluoroethylene	4	4	4	2	2	2	2	2	2	2	2	2
FEP Teflon	4	4	4	2	2	2						
PFA Teflon	4	4	4	2	2	2						
Kel-F-81 CTFE	4	4	4	2	2	2	2	2	2	2	2	2
Rulon	4	4	4	2	2	2						
Kevlar	4	4	4	2	2	2						
Carbon (CDJ-83)	4	4	4	2	2	2						
Carbon (CJPS)	4	4	4	2	2	2						
Krytox	2	2	2	2	2	2						
Fluorosilicone FS 3451	2	2	2	2	2	2						
AF-E-124 (DuPont ECD-006)	4	4	4	2	2	2						
Viton (MIL-R-83248 Class 1)	4	4	4	2	2	2						
Viton (MIL-R-83248 Class 2)	4	4	4	2	2	2	2	2	2	2	2	2
Silastic LS-53	4	4	4	2	2	2						
Polypropylene	-	-	-	2	2	2						
Dry Powder TFE (MS-122)	-	-	-	1	1	1						

TABLE 2.2-7

DATA INDICATIVE OF THE COMPATIBILITY OF LIQUID/VAPOR PHASE NITROGEN TRIFLUORIDE AT 195 K (-78 C) WITH VARIOUS NON-METALLIC MATERIALS

Material	Exposure Time, days	Type of Exposure	Specimen Surface Area, cm^2	Specimen Weights, gm Initial	Specimen Weights, gm Final	Specimen Weights, gm Change	Percent Change	Modulus of Rigidity, psi Initial	Modulus of Rigidity, psi Final	Test No.	Observations
Polytetra-fluoroethylene	33	Liquid	12.89	2.6262	2.6289	0.0027	.10	31,580	31,440	A8X	No apparent reaction
	33	Liquid	12.92	2.6498	2.6529	0.0031	.12	± 4,040	33,100	A8X	No apparent reaction
	33	Vapor	12.88	2.6366	2.6400	0.0034	.13		29,480	A8X	No apparent reaction
	33	Vapor	12.96	2.6388	2.6418	0.0030	.11		33,750	A8X	No apparent reaction
	90	Liquid	12.91	2.6328	2.6344	0.0016	.061		25,610	A8Y	No apparent reaction
	90	Liquid	12.92	2.6349	2.6349	0.0012	.046		23,930	A8Y	No apparent reaction
	90	Vapor	12.98	2.6703	2.6715	0.0012	.045		24,600	A8Y	No apparent reaction
	90	Vapor	12.99	2.6682	2.6701	0.0019	.071		26,080	A8Y	No apparent reaction
	273	Liquid	13.05	2.6726	2.6738	0.0012	.045		24,760	A8Z	No apparent reaction
	273	Liquid	13.03	2.6447	2.6462	0.0015	.057		23,970	A8Z	No apparent reaction
	273	Vapor	12.97	2.6562	2.6574	0.0012	.045		22,160	A8Z	No apparent reaction
	273	Vapor	12.96	2.6450	2.6466	0.0016	.060		20,460	A8Z	No apparent reaction
FEP Teflon	33	Liquid	13.06	2.6229	2.6234	0.0005	.019	26,310	25,000	A8X	No apparent reaction
	33	Liquid	12.95	2.6120	2.6125	0.0005	.019	± 1,800	24,460	A8X	No apparent reaction
	33	Vapor	12.94	2.6188	2.6192	0.0004	.015		24,550	A8X	No apparent reaction
	33	Vapor	13.05	2.6222	2.6227	0.0005	.019		24,910	A8X	No apparent reaction
	90	Liquid	13.10	2.6188	2.6196	0.0008	.031		26,550	A8Y	No apparent reaction
	90	Liquid	13.00	2.6140	2.6147	0.0007	.027		25,710	A8Y	No apparent reaction
	90	Vapor	13.01	2.61-5	2.6161	0.0006	.023		25,480	A8Y	No apparent reaction
	90	Vapor	13.01	2.6192	2.6199	0.0007	.027		24,650	A8Y	No apparent reaction
	273	Liquid	12.97	2.6222	2.6250	0.0028	.11		26,820	A8Z	No apparent reaction
	273	Liquid	13.01	2.6246	2.6274	0.0028	.11		26,210	A8Z	No apparent reaction
	273	Vapor	12.99	2.6177	2.6207	0.0030	.11		27,700	A8Z	No apparent reaction
	273	Vapor	12.94	2.6156	2.6186	0.0030	.11		25,690	A8Z	No apparent reaction
PFA Teflon	33	Liquid	12.98	2.6194	2.6214	0.0020	.076	25,520	25,350	A8X	No apparent reaction
	33	Liquid	12.92	2.6198	2.6218	0.0020	.076	± 1,650	24,140	A8X	No apparent reaction
	33	Vapor	12.93	2.6269	2.6288	0.0019	.072		24,270	A8X	No apparent reaction
	33	Vapor	12.82	2.5816	2.5837	0.0020	.077		25,900	A8X	No apparent reaction
	90	Liquid	12.79	2.5720	2.5734	0.0014			25,490	A8Y	No apparent reaction
	90	Liquid	12.83	2.5716	2.5728	0.0012	.054		25,580	A8Y	No apparent reaction
	90	Vapor	12.89	2.5889	2.5902	0.0013	.050		25,290	A8Y	No apparent reaction
	90	Vapor	13.08	2.7395	2.7405	0.0010	.036		24,550	A8Y	No apparent reaction
	273	Liquid	13.00	2.6314	2.6341	0.0027	.10		24,060	A8Z	No apparent reaction
	273	Liquid	12.96	2.6122	2.6151	0.0029	.11		23,830	A8Z	No apparent reaction
	273	Vapor	12.97	2.6096	2.6126	0.0030	.11		25,050	A8Z	No apparent reaction
	273	Vapor	12.86	2.5909	2.5935	0.0026	.10		24,690	A8Z	No apparent reaction
Kel-F-81 CTFE	34	Liquid	10.42	1.2854	1.2865	0.0011	.086	93,640	97,580	A9X	No apparent reaction
	34	Liquid	10.30	1.2861	1.2862	0.0011	.086	± 3,670	95,320	A9X	No apparent reaction
	34	Vapor	10.32	1.2872	1.2872	-0-	-0-		92,250	A9X	No apparent reaction
	34	Vapor	10.30	1.2873	1.2873	-0-	-0-		94,820	A9X	No apparent reaction
	90	Liquid	10.30	1.2866	1.2868	0.0002	.016		91,280	A9Y	No apparent reaction
	90	Liquid	10.36	1.2901	1.2912	0.0011	.085		92,040	A9Y	No apparent reaction
	90	Vapor	10.45	1.2871	1.2873	0.0002	.016		92,320	A9Y	No apparent reaction
	90	Vapor	10.38	1.2837	1.2868	0.0001	.008		94,490	A9Y	No apparent reaction
	272	Liquid	10.25	1.2879	1.2881	0.0002	.016		91,070	A9Z	Slight darkening of original color
	272	Liquid	10.27	1.2910	1.2912	0.0002	.016		91,150	A9Z	Slight darkening of original color

TABLE 2.2-7 (cont.)

Material	Exposure Time, days	Type of Exposure	Specimen Surface Area, cm^2	Specimen Weights, gm Initial	Specimen Weights, gm Final	Specimen Weights, gm Change	Percent Change	Modulus of Rigidity, psi Initial	Modulus of Rigidity, psi Final	Test No.	Observations
	272	Vapor	10.32	1.2905	1.2907	0.0002	.015		89,460	A9Z	Slight darkening of original color
	272	Vapor	10.29	1.2859	1.2861	0.0002	.016		90,360	A9Z	Slight darkening of original color
Rulon	33	Liquid	12.95	2.7406	2.7412	0.0006	.022	51,090	45,090	A8X	No apparent reaction
	33	Liquid	12.98	2.7457	2.7463	0.0006	.022	± 8,500	49,990	A8X	No apparent reaction
	33	Vapor	12.96	2.7304	2.7305	0.0001	.004		44,370	A8X	No apparent reaction
	33	Vapor	13.06	2.7488	2.7487	-0.0001	-.004		51,560	A8X	No apparent reaction
	90	Liquid	13.08	2.7395	2.7405	0.0010	.037		49,890	A8Y	No apparent reaction
	90	Liquid	13.03	2.7222	2.7231	0.0009	.033		46,540	A14Y	No apparent reaction
	90	Vapor	12.94	2.7308	2.7318	0.0010	.037		43,230	A14Y	No apparent reaction
	90	Vapor	12.89	2.7150	2.7159	0.0009	.033		55,130	A14Y	No apparent reaction
	275	Liquid	12.98	2.7470	2.7478	0.0008	.029		59,480	A8Z	Some color change - lighter
	275	Liquid	12.99	2.7408	2.7414	0.0006	.022		56,340	A8Z	Some color change - lighter
	275	Vapor	13.03	2.7334	2.7340	0.0006	.033		58,030	A8Z	Some color change - lighter
	275	Vapor	13.11	2.7430	2.7436	0.0006	.022		63,880	A8Z	Some color change - lighter
Kevlar	35	Liquid	NA	0.2314	0.2290	-0.0024	-1.04	NA	NA	A14X	No apparent reaction
	35	Liquid		0.2488	0.2454	-0.0034	-1.37			A14X	No apparent reaction
	35	Vapor		0.2176	0.2200	0.0024	1.10			A14X	No apparent reaction
	35	Vapor		0.2466	0.2497	0.0031	1.26			A14X	No apparent reaction
	93	Liquid		0.2443	0.2419	-0.0024	-.98			A14Y	No apparent reaction
	93	Liquid		0.2440	0.2406	-0.0034	-1.39			A14Y	No apparent reaction
	93	Vapor		0.2359	0.2387	0.0028	1.19			A14Y	No apparent reaction
	93	Vapor		0.2449	0.2461	0.0012	.49			A14Y	No apparent reaction
	275	Liquid		0.2528	0.2540	0.0012	.47			A14Z	Very slight darkening of color
	275	Liquid		0.2520	0.2532	0.0012	.48			A14Z	Very slight darkening of color
	275	Vapor		0.2496	0.2545	0.0049	1.96			A14Z	Very slight darkening of color
	275	Vapor		0.2482	0.2553	0.0071	2.86			A14Z	Very slight darkening of color
Carbon CDJ-83	31	Liquid	8.33	1.4332	1.4319	-0.0013	-.090			A10X	No apparent reaction
	31	Liquid	8.38	1.4397	1.4382	-0.0015	-.10			A10X	No apparent reaction
	31	Vapor	8.33	1.4028	1.4015	-0.0013	-.093			A10X	No apparent reaction
	31	Vapor	8.57	1.4673	1.4660	-0.0013	-.086			A10X	No apparent reaction
	92	Liquid	8.62	1.3820	1.3845	0.0025	.18			A10Y	No apparent reaction
	92	Liquid	8.32	1.4208	1.4226	0.0016	.11			A10Y	No apparent reaction
	92	Vapor	8.67	1.4414	1.4434	0.0020	.14			A10Y	No apparent reaction
	92	Vapor	8.51	1.4466	1.4488	0.0022	.15			A10Y	No apparent reaction
	273	Liquid	8.43	1.4321	1.4319	-0.0002	-.014			A10Z	No apparent reaction
	273	Liquid	8.23	1.3776	1.3778	-0.0002	-.015			A10Z	No apparent reaction
	273	Vapor	8.12	1.4197	1.4201	0.0004	.028			A10Z	No apparent reaction
	273	Vapor	8.40	1.4367	1.4370	0.0003	.020			A10Z	No apparent reaction

TABLE 2.2-7 (cont.)

Material	Exposure Time, days	Type of Exposure	Specimen Surface Area, cm^2	Specimen Weights, gm Initial	Final	Change	Percent Change	Modulus of Rigidity, psi Initial	Final	Test No.	Observations
Carbon CJPS	34	Liquid	8.44	1.4119	1.4110	-0.0009	-.063	NA	NA	A11X	No apparent reaction
	34	Liquid	8.40	1.4248	1.4237	-0.0011	-.077			A11X	No apparent reaction
	34	Vapor	8.41	1.4752	1.4743	-0.0009	-.061			A11X	No apparent reaction
	34	Vapor	8.31	1.4405	1.4396	-0.0009	-.062			A11X	No apparent reaction
	92	Liquid	8.21	1.4385	1.4386	0.0001	.007			A11Y	No apparent reaction
	92	Liquid	8.45	1.4241	1.4241	-0-	0			A11Y	No apparent reaction
	92	Vapor	8.25	1.4170	1.4169	-0.0001	-.007			A11Y	No apparent reaction
	92	Vapor	8.13	1.4233	1.4232	-0.0001	-.007			A11Y	No apparent reaction
	270	Liquid	8.29	1.4332	1.4332	-0-	0			A11Z	No apparent reaction
	270	Liquid	8.36	1.4475	1.4478	0.0002	.021			A11Z	No apparent reaction
	270	Vapor	8.53	1.4575	1.4580	0.0005	.034			A11Z	No apparent reaction
	270	Vapor	8.53	1.4099	1.4109	0.0010	.071			A11Z	No apparent reaction
Krytox	33	Liquid	NA	0.1810	0.0060	-0.1750	-96.7			A12X	Nearly all dispersed
	33	Vapor		1.2468	0.0068	-1.2400	-99.5			A12X	Nearly all dispersed
	273	Liquid		1.2918	-0-	-0-	-100			A12Z	Nearly all dispersed
	273	Liquid		0.2554	-0-	-0-	-100			A12Z	Nearly all dispersed
Krytox, Vacuum-Stripped	32	Liquid	NA	0.9493	-0-	-0-	-100			A12*X	All dispersed
	32	Liquid		0.4887	-0-	-0-	-100			A12*X	All dispersed
	32	Vapor		1.0004	-0-	-0-	-100			A12*X	All dispersed
	32	Vapor		0.9138	-0-	-0-	-100			A12*X	All dispersed
Fluorosilicone FS3451	33	Liquid	NA	1.1929	-0-	-1.1929	-100			A13X	Specimen dispersed
	33	Vapor		1.1020	-0-	-1.1020	-100			A13X	Specimen dispersed
	91	Liquid		1.4243	-0-	-1.4243	-100			A13Y	Specimen dispersed
	91	Vapor		1.3752	-0-	-1.3752	-100			A13Y	Specimen dispersed
	273	Liquid		1.3951	-0-	-1.3951	-100			A13Z	Specimen dispersed
	273	Vapor		1.5599	-0-	-1.5599	-100			A13Z	Specimen dispersed

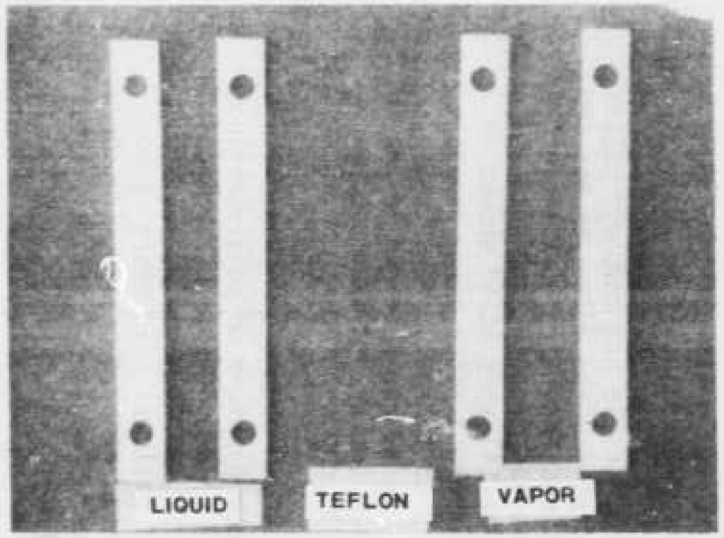
weight and appearance changes, the modulus of rigidity values are given for the thermoplastics.

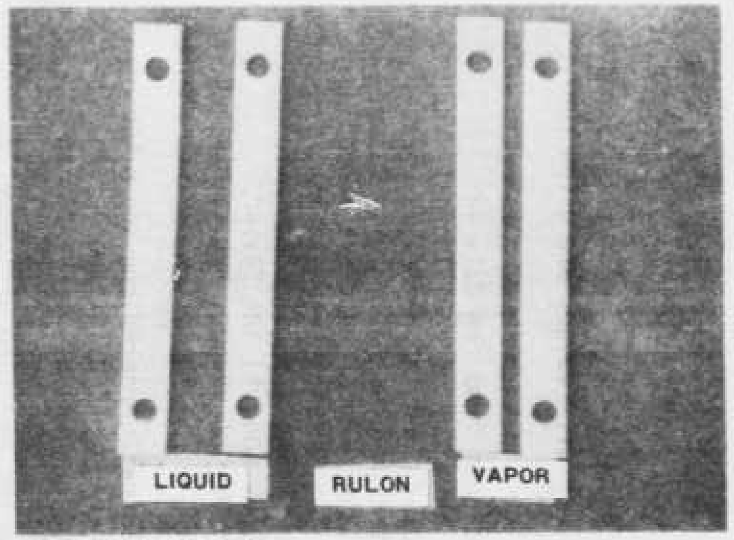
The significant items to note from the data in Table 2.2-7 are as follows: (1) the greases, both Krytox and Fluorosilicone FS 3451, dissolved readily in nitrogen trifluoride and the materials were dispersed throughout the test container; (2) the modulus of rigidity values of the thermoplastics were unaffected by the liquid/vapor exposure except for polytetrafluoroethylene which exhibited a significant decrease in values with increasing exposure times; (3) the graphite samples exhibited weight changes of about 0.1% which is acceptable, and (4) the Kevlar exhibited a slight color change and a variable weight change, but maintained its structural integrity.

Photographs of the polytetrafluoroethylene and Rulon specimens after 9 months exposure are shown in Figure 2.2.12; photographs of the FEP and PFA Teflon specimens after 9-months exposure are shown in Figure 2.2.13; the Kel-F specimens after 9-months exposure are shown in Figure 2.2.14; the carbon specimens after 9 months exposure are shown in Figure 2.2.15; and the Kevlar specimens after 9-months exposure is shown in Figure 2.2.16.

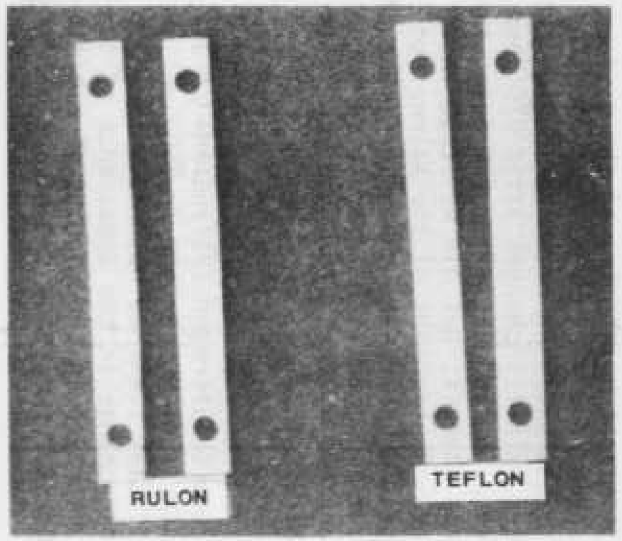
The data obtained from testing the elastomeric materials in both liquid- and vapor-phase nitrogen trifluoride are presented in Table 2.2-8. The significant items to note from the data are as follows: (1) Kalrez was visibly affected by the nitrogen trifluoride as evidenced by the slight blistering of the surface and the tensile and hardness values did decrease with increasing periods of exposure; (2) the Vitons except for the slight swelling noted for two of the specimens exhibited no visible changes, minimal changes after 270 days in tensile strength, and some decrease in hardness after 270 days; and (3) the Silastic LS-53 showed no visible changes, but did decrease in tensile strength after every exposure period. Photographs of the Kalrez and Silastic LS-53 specimens after 9-months exposure are shown in Figure 2.2.17. A Kalrez specimen in the as-received condition is shown in the photograph as the untagged specimen. Photographs of the Viton specimens after 9-month exposure are shown in Figure 2.2.18.

The data obtained from the exposure of polytetrafluoroethylene to nitrogen trifluoride at 344 K (160 F) and pressures ranging from 3.45 to 17.24 MN/m^2 (500 to 2500 psia) are presented in Table 2.2-9. The significant items to note from the data are as follows: (1) there is no visible change in the appearance of the specimens after exposure, (2) there is a decrease in the modulus of rigidity values which is not entirely time dependent, (specimens from Test No. D8X were reused in Test *DNX giving

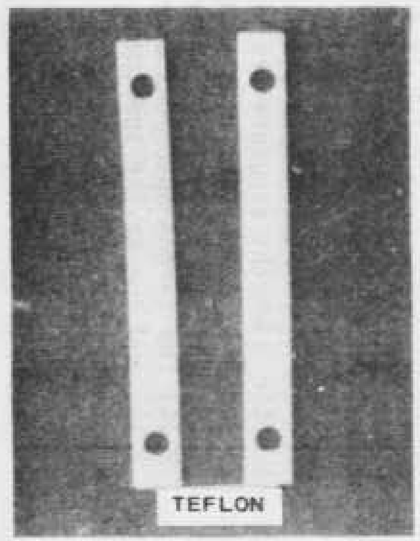




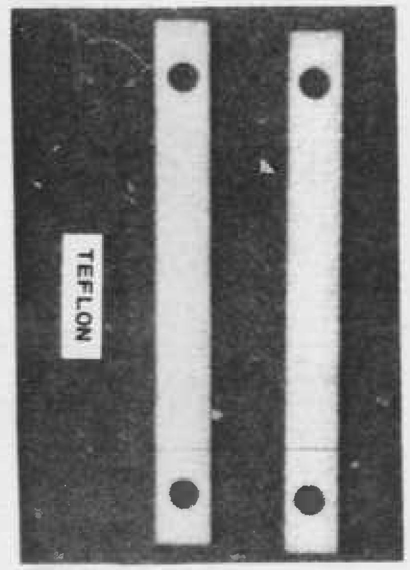
Conditions: Liquid/Vapor, 195°K (-108°F)



Conditions: 3.45 MN/m^2 (500 psia), 344°K (160°F)

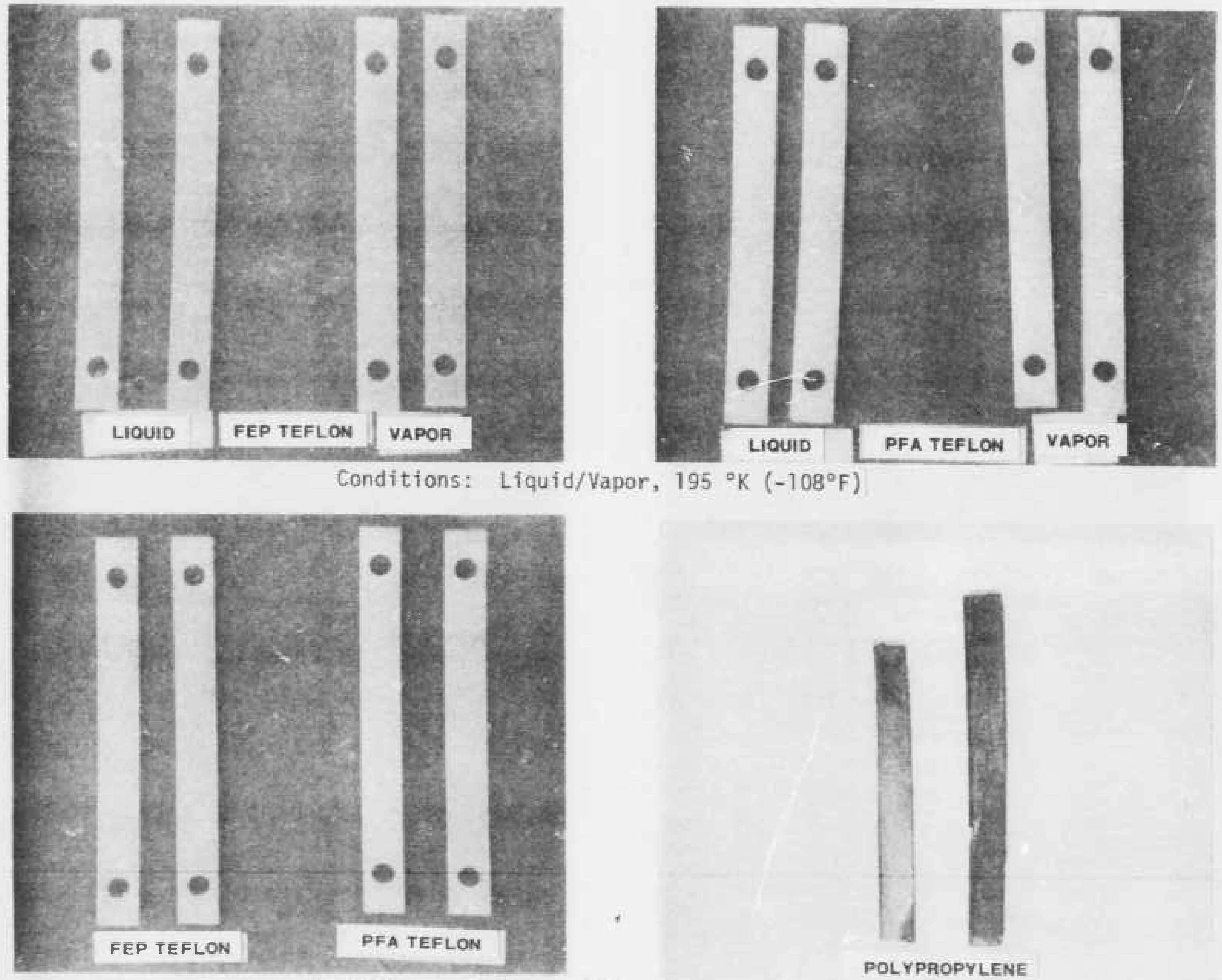


Conditions: 10.34 MN/m^2 (1500 psia), 344°K (160°F)



Conditions: 17.24 MN/m^2 (2500 psia), 344°K (160°F)

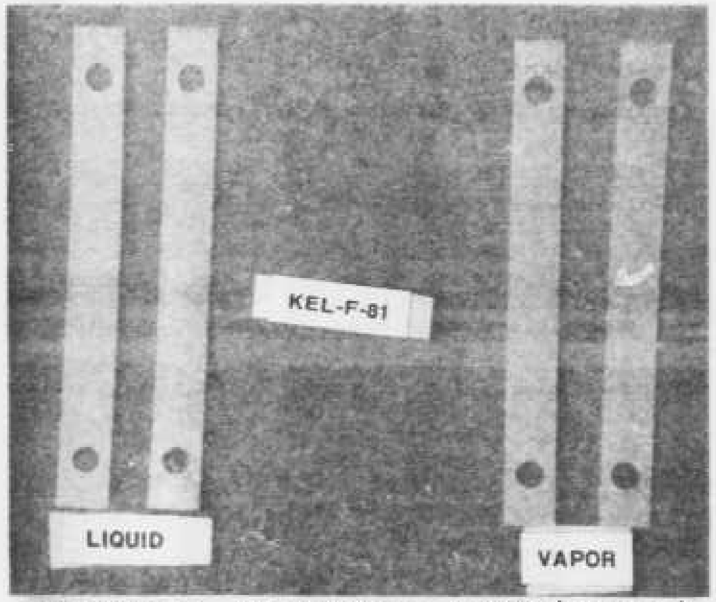
Figure 2.2.12. Polytetrafluoroethylene and Rulon Specimens After 9 Months Static Exposure to Nitrogen Trifluoride



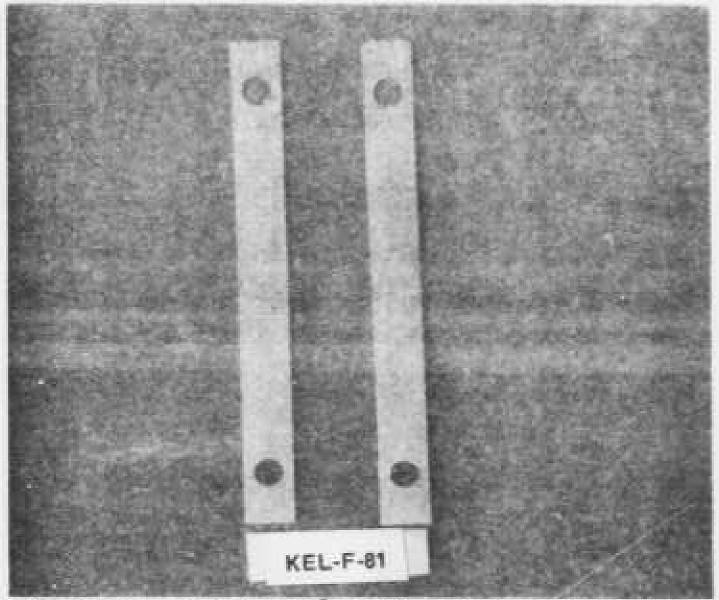
Conditions: Liquid/Vapor, 195 °K (-108°F)

Conditions: 3.45 MN/m^2 (500 psia), 344 °K (160°F)

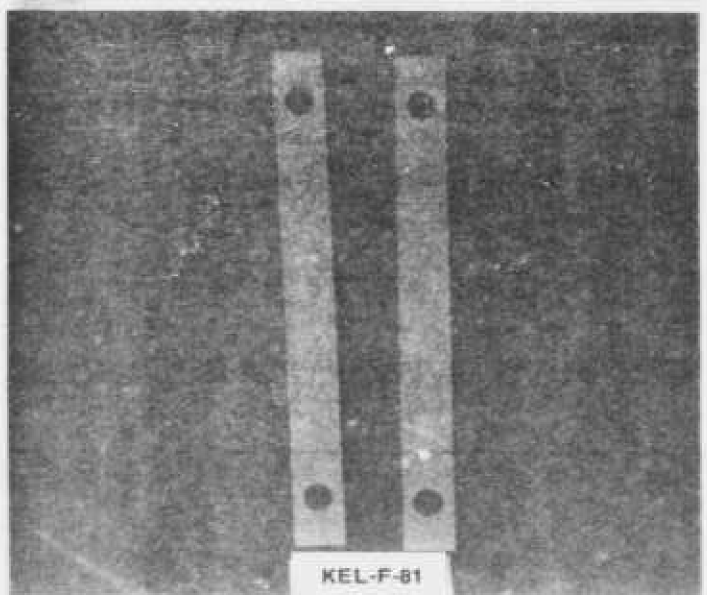
Figure 2.2.13. FEP Teflon, PFA Teflon, and Polypropylene Specimens After 9 Months Static Exposure to Nitrogen Trifluoride



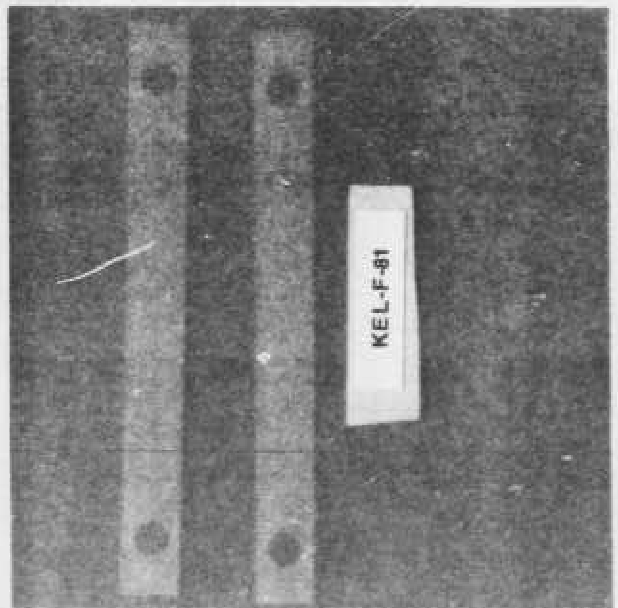
Conditions: Liquid/Vapor, 195°K (-108 °F)



Conditions: 3.45 MN/m^2 (500 psia), 344 °K (160°F)

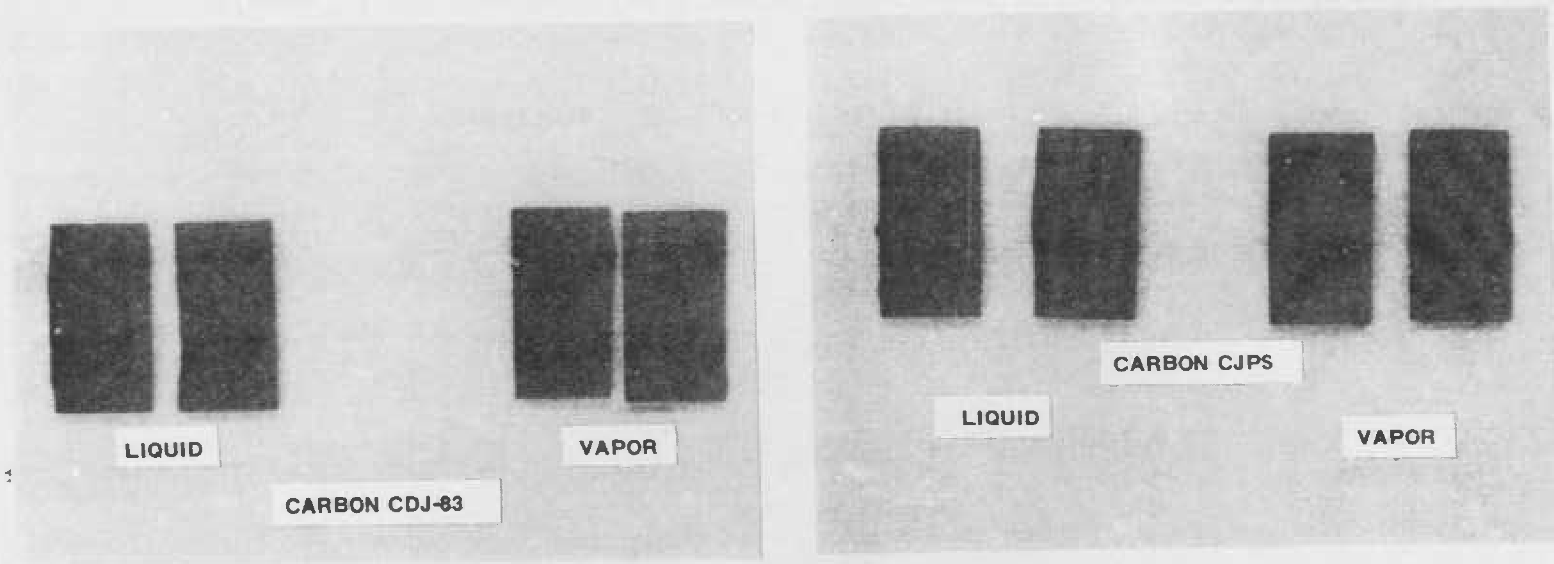


Conditions: 10.34 MN/m^2 (1500 psia), 344 °K (160°F)

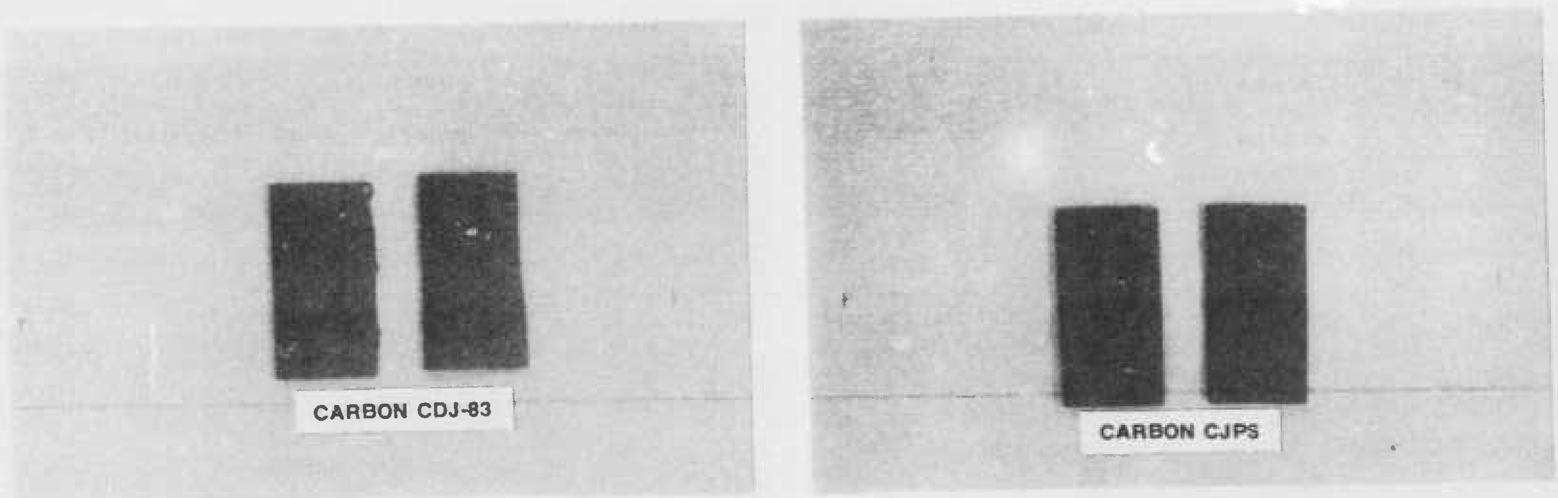


Conditions: 17.24 MN/m^2 (2500 psia), 344°K (160°F)

Figure 2.2.14. Kel-F 81 Specimens After 9 Months Static Exposure to Nitrogen Trifluoride

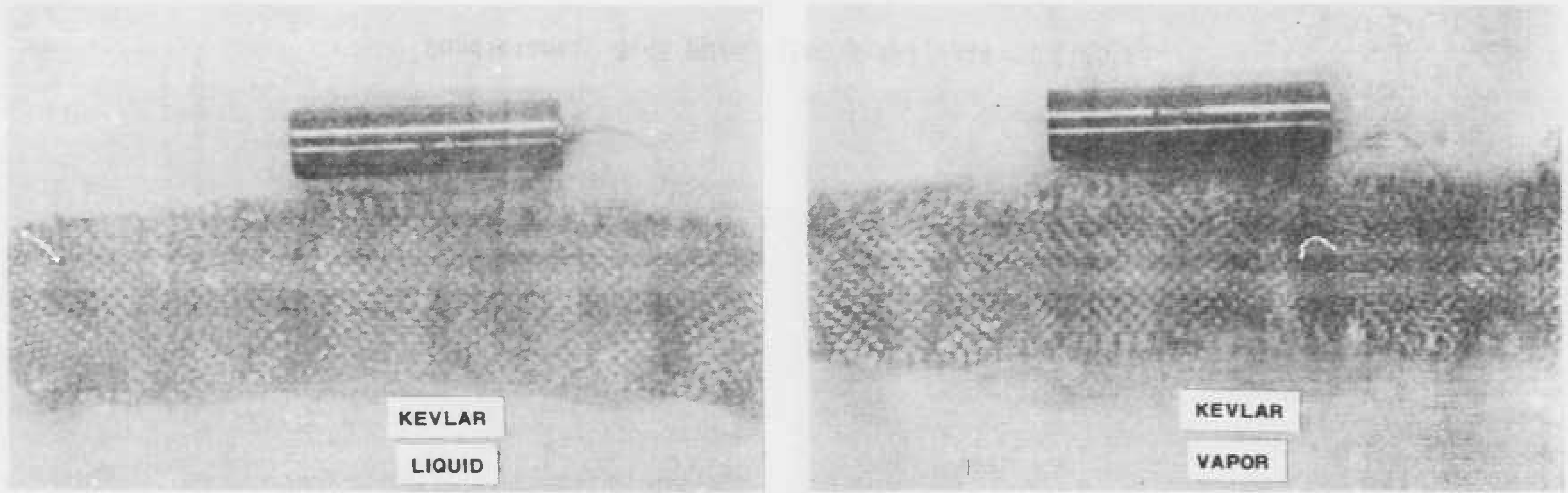


Conditions: Liquid/Vapor, 195°K (-108°F)

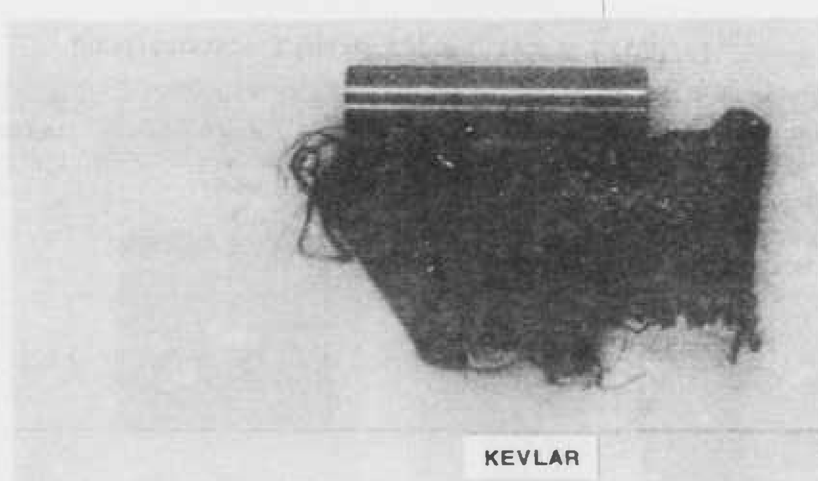


Conditions: 3.45 MN/m_2 (500 psia), 344 °K (160°F)

Figure 2.2.15. Carbon Specimens After 9 Months Static Exposure to Nitrogen Trifluoride



Conditions: Liquid/Vapor, 195°K (-108°F)



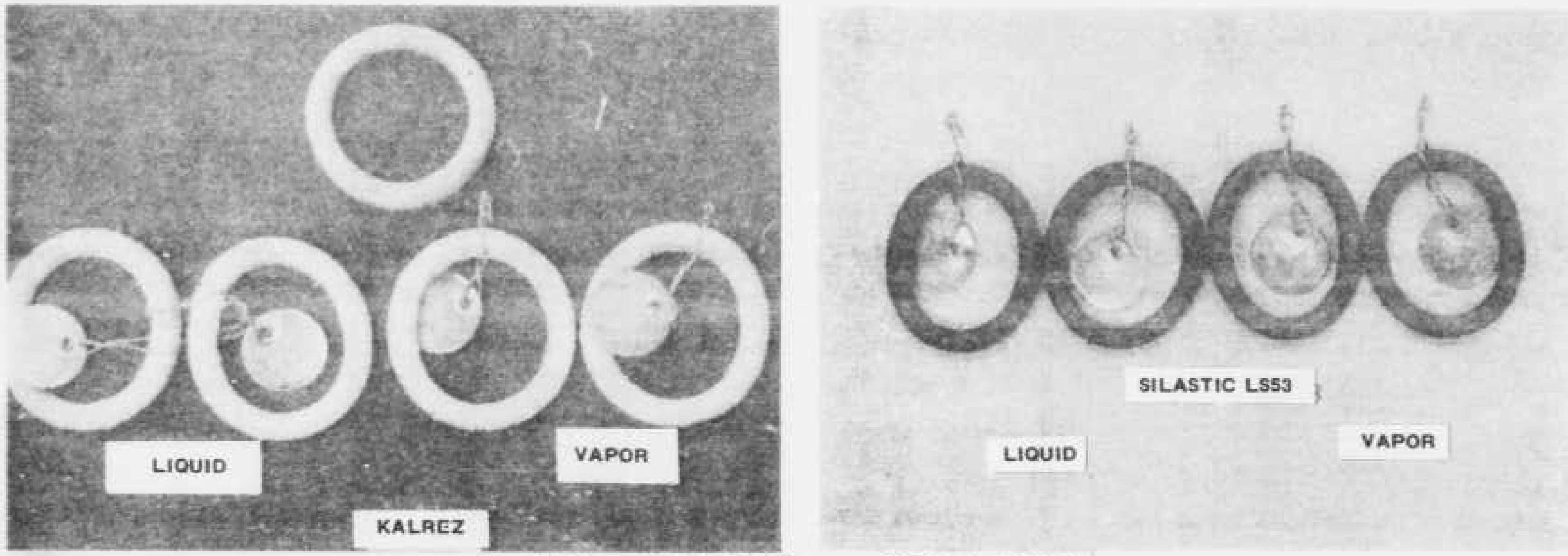
Conditions: 3.45 MN/m^2 (500 psia), 344°K (160°F)

Figure 2.2.16. Kevlar After 9 Months Static Exposure to Nitrogen Trifluoride

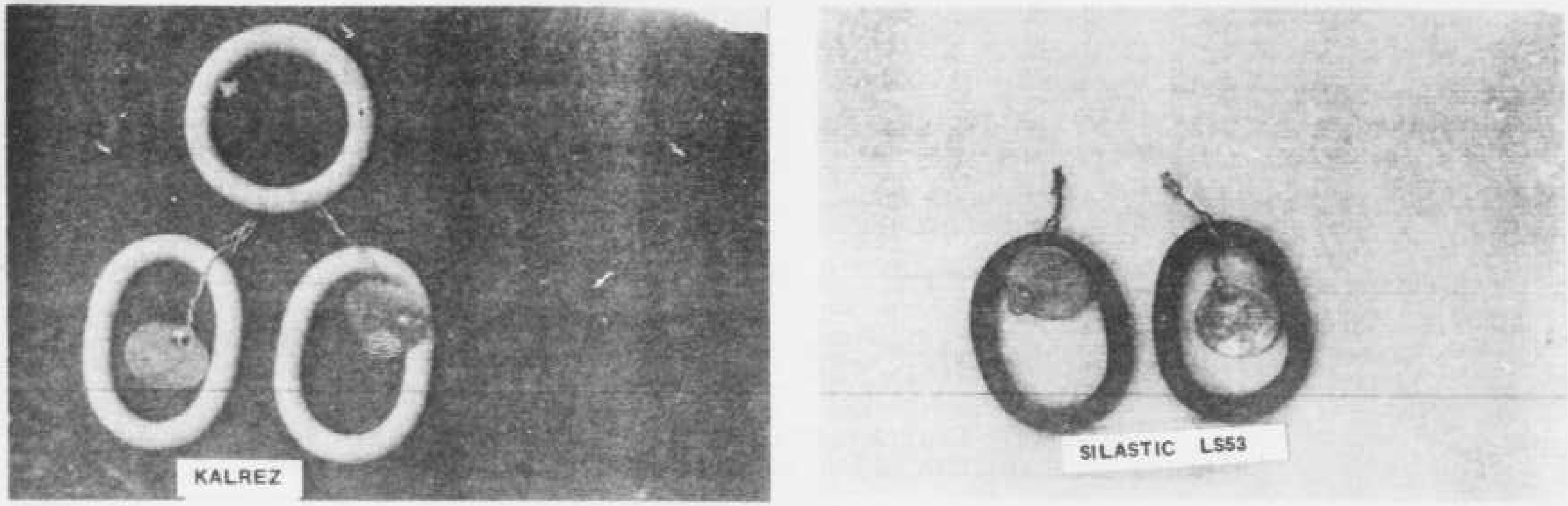
TABLE 2.2-8

DATA INDICATIVE OF THE COMPATIBILITY OF LIQUID/VAPOR PHASE NITROGEN TRIFLUORIDE AT 195 K (-78 C) WITH VARIOUS ELASTOMERS

Material and Test No.	Exposure Time, days	Type of Exposure	Specimen Surface Area, cm^2	Specimen Weights, gm			Tensile Strength, psi		Modulus at 100% Elongation, psi		Ultimate Elongation, %		Wallace Hardness		Observations
				Initial	Final	Change	Initial	Final	Initial	Final	Initial	Final	Initial	Final	
Kalrez, A15X	33	Liquid	8.67	1.5283	1.5451	0.0168	1778	1543	694	690	173	165	68	67	Very white coloration, slight blistering
	33	Liquid	8.68	1.5299	1.5464	0.0165	± 119	1631	± 38	643	± 17	174	± 0.5	67	Very white coloration, slight blistering
	33	Vapor	8.69	1.5299	1.5388	0.0089		1762		642		174		68	Very white coloration, slight blistering
	33	Vapor	8.71	1.5330	1.5490	0.0160		1558		701		157		68	Very white coloration, slight blistering
A15Y	91	Liquid	8.88	1.5317	1.5328	0.0011		1560		668		156		68	No apparent reaction
	91	Liquid	8.91	1.5331	1.5344	0.0013		1698		742		165		68	No apparent reaction
	91	Vapor	8.82	1.5230	1.5238	0.0008		1322		763		148		67	No apparent reaction
	91	Vapor	8.96	1.5366	1.5375	0.0009		1618		758		156		67	No apparent reaction
A15Z	274	Liquid	8.85	1.5321	1.5453	0.0132		1255		760		130		53	Very white, surface bubbled
	274	Liquid	8.83	1.5200	1.5331	0.0131		1327		790		139		50	Very white, surface bubbled
	274	Vapor	8.94	1.5335	1.5450	0.0115		748		748		96		58	Very white, surface bubbled
	274	Vapor	8.85	1.5251	1.5366	0.0115		1028		717		130		56	Very white, surface bubbled
Viton, Class I	33	Liquid	8.79	1.2316	1.2325	0.0009	1394	1143	453	520	230	178	74	74	No apparent reaction
	33	Liquid	8.79	1.2264	1.2276	0.0012	± 44	1391	± 23	491	± 16	227	± 0.5	70	No apparent reaction
A17X	33	Vapor	8.79	1.2292	1.2301	0.0009		975		471		166		73	No apparent reaction
	33	Vapor	8.79	1.2186	1.2195	0.0009		1170		488		192		72	No apparent reaction
A17Y	91	Liquid	8.66	1.2117	1.2120	0.0003		1199		421		209		72	No apparent reaction
	91	Liquid	8.79	1.2296	1.2301	0.0005		1118		425		192		72	No apparent reaction
	91	Vapor	8.79	1.2188	1.2192	0.0004		1337		511		209		73	No apparent reaction
	91	Vapor	8.66	1.2096	1.2097	0.0001		1301		590		174		74	No apparent reaction
A17Z	270	Liquid	8.72	1.2272	1.2287	0.0015		1316		699		166		65	No apparent reaction
	270	Liquid	8.70	1.2237	1.2253	0.0016		1195		556		183		71	No apparent reaction
	270	Vapor	8.65	1.2181	1.2196	0.0015		1352		561		209		69	No apparent reaction
	270	Vapor	8.63	1.2184	1.2200	0.0016		1362		598		180		60	No apparent reaction
Viton, Class II	33	Liquid	9.04	1.2800	1.2805	0.0005	1483	1059	992	1059	167	100	92	93	No apparent reaction
	33	Liquid	9.17	1.2964	1.2969	0.0005	± 107	1222	± 20	983	± 14	134	± 0.8	90	No apparent reaction
A17X	33	Vapor	9.04	1.2819	1.2823	0.0004		1417		1044		156		93	Slightly swollen
	33	Vapor	9.04	1.2856	1.2859	0.0003		1075		997		113		91	Slightly swollen
A17Y	91	Liquid	9.23	1.2900	1.2899	-0.0001		1235		906		139		91	No apparent reaction
	91	Liquid	9.04	1.2705	1.2702	-0.0003		1547		966		174		91	No apparent reaction
	91	Vapor	9.04	1.2693	1.2691	-0.0002		1399		800		166		91	No apparent reaction
	91	Vapor	9.10	1.2911	1.2911	-0-		1449		1036		166		91	No apparent reaction
A17Z	270	Liquid	9.00	1.3019	1.3030	0.0011		1417		1215		131		81	No apparent reaction
	270	Liquid	9.04	1.2981	1.2992	0.0011		1366		1160		122		82	No apparent reaction
	270	Vapor	9.03	1.2761	1.2771	0.0010		1604		1303		122		73	No apparent reaction
	270	Vapor	9.03	1.2753	1.2764	0.0009		1501		1160		148		73	No apparent reaction
Silastic LS-53	33	Liquid	8.58	0.9534	0.9554	0.0020	661	499	361	396	159	122	63	74	No apparent reaction
	33	Liquid	8.50	0.9534	0.9550	0.0016	± 124	312	± 13	---	± 25	78	± 0.6	72	No apparent reaction
A16X	33	Vapor	8.60	0.9539	0.9548	0.0009		513		427		114		73	No apparent reaction
	33	Vapor	8.58	0.9506	0.9516	0.0010		388		382		105		73	No apparent reaction
A16Y	91	Liquid	8.62	0.9511	0.9531	0.0020		355		---		96		64	No apparent reaction
	91	Liquid	8.61	0.9532	0.9551	0.0019		554		435		131		65	No apparent reaction
	91	Vapor	8.55	0.9535	0.9553	0.0018		619		387		148		64	No apparent reaction
	91	Vapor	8.66	0.9550	0.9570	0.0020		386		---		96		65	No apparent reaction
A16Z	270	Liquid	8.58	0.9535	0.9537	0.0002		351		---		78		61	No apparent reaction
	270	Liquid	8.51	0.9518	0.9520	0.0002		510		452		113		64	No apparent reaction
	270	Vapor	8.61	0.9556	0.9551	-0.0005		460		435		105		62	No apparent reaction
	270	Vapor	8.69	0.9543	0.9541	-0.0002		527		435		122		59	No apparent reaction



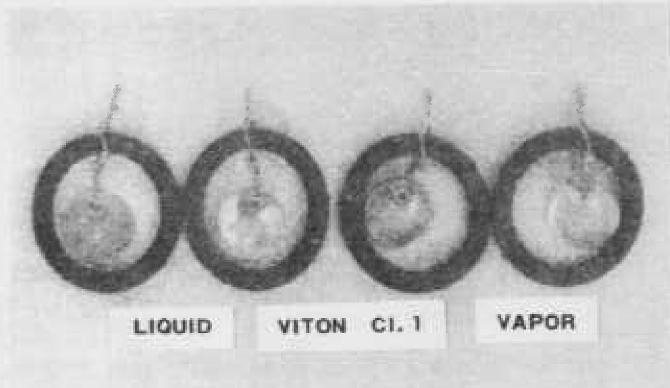
Conditions: Liquid/Vapor, 195°K (-108°F)

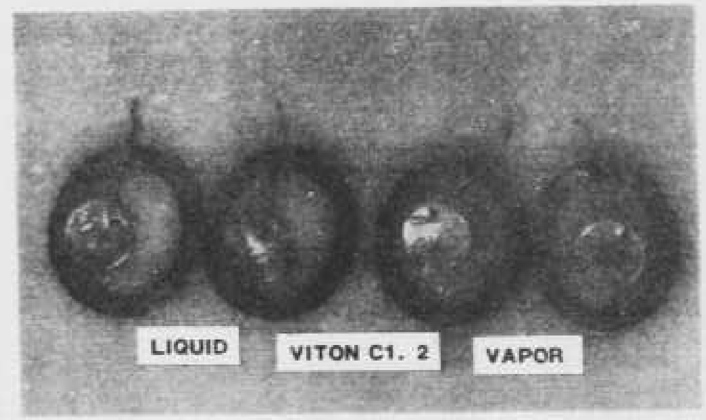


Conditions: 3.45 MN/m^2 (500 psia), 344°K (160°F)

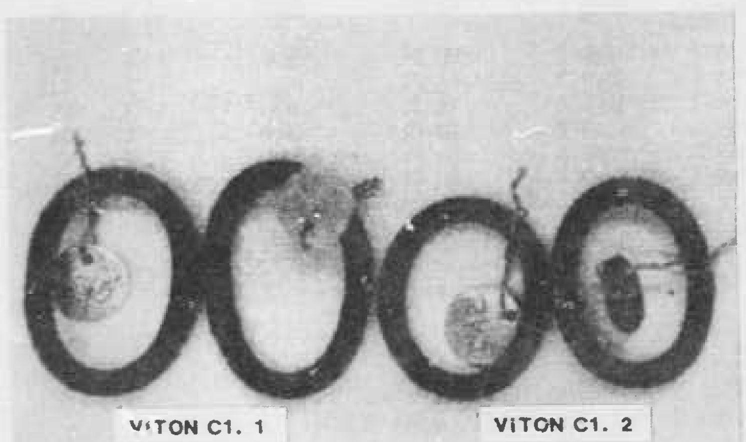
Figure 2.2.17. Kalrez (Dupont ECD-006) and Silastic LS-53 Specimens After 9 Months Static Exposure to Nitrogen Trifluoride

Note: Unexposed Kalrez specimen in upper portion of each group for comparison purposes





Conditions: Liquid/Vapor, 195°K (-108°F)



Conditions: 3.45 MN/m^2 (500 psia), 344°K (160°F)

Conditions:
10.34 MN/m^2 (1500 psia),
344°K (160°F)

Conditions:
17.24 MN/m^2 (2500 psia),
344°K (160°F)

Figure 2.2.18. Viton Specimens After 9 Months Static Exposure to Nitrogen Trifluoride

TABLE 2.2-9

DATA INDICATIVE OF THE COMPATIBILITY OF GASEOUS NITROGEN TRIFLUORIDE AT 344 K (160 F) AND PRESSURES RANGING FROM 3.45 to 17.24 MN/m^2 (500 TO 2500 PSIA) WITH POLYTETRAFLUOROETHYLENE

Exposure Conditions			Specimen Surface Area	Specimen Weights, gm			Percent	Modulus of Rigidity, psi		Test	
Time, Days	MN/m^2	Pressure psia	cm^2	Initial	Final	Change	Change	Initial	Final	No.	Observation
30	3.45	500	13.08	2.6781	2.6887	0.0106	0.40	31,580	29,350	B8X	No apparent reaction.
30	3.45	500	13.02	2.6476	2.6584	0.0108	0.41	±4,040	31,720	B8X	No apparent reaction.
90	3.45	500	13.03	2.6619	2.6649	0.0030	0.11		27,930	B8Y	No apparent reaction.
90	3.45	500	12.91	2.6440	2.6474	0.0034	0.13		27,840	B8Y	No apparent reaction.
272	3.45	500	12.99	2.6400	2.6421	0.0021	0.080		26,140	B8Z	No apparent reaction.
272	3.45	500	12.96	2.6418	2.6438	0.0020	0.076		24,740	B8Z	No apparent reaction.
30	8.62	1250	13.03	2.6594	2.6720	0.0126	0.47		28,500	C8X	No apparent reaction.
30	8.62	1250	13.02	2.6648	2.6791	0.0143	0.54		29,540	C8X	No apparent reaction.
30	13.44	1950	12.94	2.6327	2.6463	0.0136	0.52		28,250	D8X	No apparent reaction.
30	13.44	1950	13.04	2.6672	2.6810	0.0138	0.52		28,710	D8X	No apparent reaction.
29	17.24	2500	13.05	2.6327	2.6332	0.0005	0.019	(28,250)	23,280	*DNX	No apparent reaction.
29	17.24	2500	13.12	2.6672	2.6676	0.0004	0.015	(28,710)	22,420	*DNX	No apparent reaction.
90	10.34	1500	12.96	2.6533	2.6567	0.0034	0.13		27,960	C8Y	No apparent reaction.
90	10.34	1500	12.93	2.6490	2.6572	0.0082	0.31		27,700	C8Y	No apparent reaction.
89	17.24	2500	12.95	2.6535	2.6538	0.0003	0.011		26,740	D8Y	No apparent reaction.
89	17.24	2500	12.91	2.6635	2.6638	0.0003	0.011		23,570	D8Y	No apparent reaction.
273	10.34	1500	13.04	2.6734	2.6764	0.0030	0.011		25,370	C8Z	No apparent reaction.
273	10.34	1500	12.90	2.6281	2.6320	0.0039	0.015		26,480	C8Z	No apparent reaction.
269	17.24	2500	12.92	2.6363	2.6368	0.0005	0.019		23,970	D8Z	No apparent reaction.
269	17.24	2500	13.04	2.6689	2.6696	0.0010	0.037		23,530	D8Z	No apparent reaction.

an accumulated exposure period of 59 days; the values after 59 days exposure are comparable to the values obtained after 269 days exposure at the 17.24 MN/m^2 (2500 psia) condition) and (3) polytetrafluoroethylene is compatible with gaseous nitrogen trifluoride under static conditions. Photographs of the specimens after the 9 month exposure period are shown in Figure 2.2.12.

The data from the exposure of Kel-F 81 CTFE to nitrogen trifluoride at 344 K (160 F) and pressures ranging from 3.45 MN/m^2 to 17.24 MN/m^2 (500 to 2500 psia) are presented in Table 2.2-10. The significant items to note from the data are as follows: (1) there is a visible change in appearance of the Kel-F in which blotches of white coloration appear within the specimens, and (2) the modulus of rigidity values for the Kel-F decrease significantly with the time of exposure as the pressure of the nitrogen trifluoride is increased to 17.24 MN/m^2 (2500 psia), (the modulus of rigidity values for the specimens used for 29 day exposure at 17.24 MN/m^2 (2500 psia) were determined prior to and after exposure) and (3) no apparent physical degradation of Kel-F occurs at the 3.45 MN/m^2 (500 psia) pressure level during the 272 days of exposure. Photographs of the specimens after the exposures are shown in Figure 2.2.14.

The data for the remaining thermoplastic materials, the graphites, the greases, and the thermosetting plastic Kevlar which were exposed to nitrogen trifluoride at 344 K (160 F) and 3.45 MN/m^2 (500 psia) are presented in Table 2.2-11.

The significant items to note from the data are as follows: (1) FEP and PFA Teflons are apparently unaffected by this exposure condition; (2) Rulon exhibits some color change but no statistically significant degradation in modulus of rigidity; (3) Kevlar undergoes color change, but maintains significant structural integrity (see Figure 2.2.16); (4) the carbons exhibit weight changes of less than 1% and the white deposit on the Carbon CDJ-83 may be due to the interaction of the phosphate salt with nitrogen trifluoride, (5) polypropylene changes significantly and is apparently unsuitable for prolonged exposure to nitrogen trifluoride, (6) the greases, Krytox and Fluorisilicone FS 3451 dispersed throughout the test container exhibiting a miscibility characteristic that is not tolerable in a use-system, and (7) the Dry Powder TFE underwent significant weight changes but no visibly apparent changes.

The data from the exposure of Kalrez, Viton, Class I, and Silastic LS 53 to nitrogen trifluoride at 344 K (160 F) and 3.45 MN/m^2 (500 psia) are presented in Table 2.2-12. The significant items to note from the data are as follows: (1) the Viton, Class I and Silastic LS 53 deteriorated to the extent that tensile properties could not be determined

TABLE 2.2-10

DATA INDICATIVE OF THE COMPATIBILITY OF GASEOUS NITROGEN TRIFLUORIDE AT 344 K (160 F) AND PRESSURES RANGING FROM 3.45 to 17.24 MN/m^2 (500 TO 2500 PSIA) WITH KEL-F-81 CTFE

Exposure Conditions			Specimen Surface Area	Specimen Weights, gm			Percent	Modulus of Rigidity, psi		Test	
Time, Days	Pressure MN/m^2	Pressure psia	cm^2	Initial	Final	Change	Change	Initial	Final	No.	Observations
32	3.45	500	10.33	1.2869	1.2886	0.0017	0.13	93,640	94,820	B9X	No apparent reaction.
32	3.45	500	10.32	1.2878	1.2895	0.0017	0.13	±3,670	97,700	B9X	No apparent reaction.
90	3.45	500	10.34	1.2894	1.2943	0.0049	0.38		91,630	B9Y	No apparent reaction.
90	3.45	500	10.28	1.2904	1.2953	0.0049	0.38		90,430	B9Y	No apparent reaction.
272	3.45	500	10.36	1.2838	1.2861	0.0023	0.18		98,170	B9Z	Blotchy white spots
272	3.45	500	10.31	1.2869	1.2893	0.0024	0.19		100,240	B9Z	Blotchy white spots
30	8.62	1250	10.33	1.2904	1.2992	0.0088	0.68		89,620	C9X	No apparent reaction.
30	8.62	1250	10.29	1.2901	1.2990	0.0089	0.69		92,770	C9X	No apparent reaction
30	13.44	1950	10.35	1.2877	1.3043	0.0166	1.29		67,990	D9X	No apparent reaction.
30	13.44	1950	10.32	1.2850	1.3027	0.0177	1.38		68,830	D9X	No apparent reaction.
29	17.24	2500	10.30	1.2851	1.2943	0.0082	0.64	(95,320)	79,740	*DNX	Blotchy white spots.
29	17.24	2500	10.32	1.2872	1.2952	0.0080	0.62	(92,250)	79,230	*DNX	Blotchy white spots.
91	10.34	1500	10.35	1.2861	1.2962	0.0101	0.78		75,490	C9Y	Whites in coloration, blotches.
91	10.34	1500	10.36	1.2868	1.2967	0.0099	0.77		78,280	C9Y	Whites in coloration, blotches.
88	17.24	2500	10.34	1.2880	1.2987	0.0107	0.83		77,570	D9Y	Significant white blotches.
88	17.24	2500	10.39	1.2862	1.2963	0.0101	0.79		73,470	D9Y	Significant white blotches.
273	10.34	1500	10.32	1.2850	1.2976	0.0126	0.98		80,840	C9Z	Blotchy white spots.
273	10.34	1500	10.29	1.2872	1.2998	0.0126	0.98		72,830	C9Z	Blotchy white spots.
269	17.24	2500	10.29	1.2864	1.3035	0.0171	1.32		42,310	D9Z	Blotchy white spots.
269	17.24	2500	10.29	1.2878	1.3048	0.0170	1.32		44,310	D9Z	Blotchy white spots.

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TABLE 2.2-11

DATA INDICATIVE OF THE COMPATIBILITY OF GASEOUS NITROGEN TRIFLUORIDE AT 344 K (160 F) AND 3.45 MN/m^2 (500 PSIA) WITH VARIOUS NON-METALLIC MATERIALS

Material	Exposure Time, days	Specimen Surface Area, cm^2	Specimen Weights, gm Initial	Specimen Weights, gm Final	Specimen Weights, gm Change	Percent Change	Modulus of Rigidity, psi Initial	Modulus of Rigidity, psi Final	Test No.	Observations
FEP Teflon	30	13.00	2.6203	2.6289	0.0086	.32	26,310	26,080	B8X	No apparent reaction
	30	13.01	2.6144	2.6279	0.0135	.52	$\pm$ 1,800	25,310	B8X	No apparent reaction
	90	12.99	2.6126	2.6158	0.0032	.12		26,020	B8Y	No apparent reaction
	90	12.94	2.6135	2.6165	0.0030	.11		25,700	B8Y	No apparent reaction
	272	12.98	2.6206	2.6219	0.0013	.050		25,280	B8Z	No apparent reaction
	272	12.96	2.6079	2.6092	0.0013	.050		24,790	B8Z	No apparent reaction
PFA Teflon	30	12.82	2.5716	2.5792	0.0076	.30	25,520	25,490	B8X	No apparent reaction
	30	12.97	2.6157	2.6241	0.0084	.32	$\pm$ 1,650	27,520	B8X	No apparent reaction
	90	13.01	2.6312	2.6326	0.0014	.053		25,210	B8Y	No apparent reaction
	90	12.84	2.5738	2.5752	0.0014	.054		26,720	B8Y	No apparent reaction
	272	12.85	2.5850	2.5858	0.0008	.031		26,500	B8Z	No apparent reaction
	272	12.93	2.6031	2.6040	0.0009	.035		23,730	B8Z	No apparent reaction
Rulon	30	12.96	2.7365	2.7419	0.0054	.20	51,090	50,640	B8X	Color change from brown to yellowish-white
	30	13.02	2.7407	2.7459	0.0052	.19	$\pm$ 8,500	61,420	B8X	Color change from brown to yellowish-white
	90	13.02	2.7426	2.7446	0.0020	.073		46,260	B8Y	No apparent reaction
	90	13.05	2.7298	2.7320	0.0020	.073		57,970	B8Y	No apparent reaction
	272	12.98	2.7425	2.7435	0.0010	.036		49,910	B8Z	Color change from tan to yellow tinge
	272	13.15	2.7681	2.7689	0.0008	.029		44,190	B8Z	Color change from tan to yellow tinge
Kevlar	32		0.2374	0.2394	0.0020	.84	N.A.	N.A.	B14X	Slight darkening of the material
	32		0.2417	0.2437	0.0020	.82			B14X	Slight darkening of the material
	92		0.2430	0.2448	0.0018	.74			B14Y	Slight darkening of the material
	92		0.2327	0.2347	0.0020	.86			B14Y	Slight darkening of the material
	274		0.2409	0.2391	-0.0018	-.75			B14Z	Reddish-brown color
	274		0.2539	0.2524	-0.0015	-.59			B14Z	Reddish-brown color
Carbon CDJ-83	32	8.57	1.4358	1.4283	-0.0075	-.52			B10X	No apparent reaction
	32	8.36	1.4376	1.4310	-0.0066	-.46			B10X	No apparent reaction
	90	8.89	1.4696	1.4702	0.0006	.041			B10Y	No apparent reaction
	90	8.55	1.4475	1.4482	0.0007	.048			B10Y	No apparent reaction
	273	8.65	1.4289	1.4254	-0.0035	-.24			B10Z	White surface deposit-easily hydrolysed
	273	8.55	1.4032	1.3896	-0.0136	-.97			B10Z	White surface deposit-easily hydrolysed
Carbon CJPS	32	8.41	1.4372	1.4413	0.0041	.29			B11X	No apparent reaction
	32	8.58	1.4359	1.4398	0.0039	.27			B11X	No apparent reaction
	91	8.18	1.4306	1.4313	0.0007	.049			B11Y	No apparent reaction
	91	8.46	1.4494	1.4499	0.0005	.034			B11Y	No apparent reaction
	273	8.37	1.4452	1.4526	0.0074	.51			B11Z	No apparent reaction
	273	8.32	1.4078	1.4150	0.0072	.51			B11Z	No apparent reaction
Krytox	32		0.8755	0.8148	-0.0607	-6.9			B12X	Some material dissolved and deposited on
	32		0.3495	0.2369	-0.1126	-32.2			B12X	container walls.
	272		0.9509	-0-	-0-	-100			B12Z	Specimen dissolved - white in color
	272		0.2113	-0-	-0-	-100			B12Z	Specimen dissolved - white in color

TABLE 2.2-11 (cont.)

Material	Exposure Time, days	Specimen Surface Area, cm^2	Specimen Weights, gm Initial	Specimen Weights, gm Final	Specimen Weights, gm Change	Percent Change	Modulus of Rigidity, psi Initial	Modulus of Rigidity, psi Final	Test No.	Observations
Krytox, Vacuum	31		0.4738	0.1224	-0.3514	-74.2	N.A.	N.A.	B12*X	Some apparent softening - most material
Stripped	31		1.1730	0.6709	-0.5021	-42.8			B12*X	on container bottom.
Fluorosilicone	30		1.4090	1.1723	-0.2367	-16.8			B13X	Some material dissolved and deposited
FS3451	30		1.1549	0.3988	-0.7561	-65.5			B13X	on container walls.
	272		1.3301	-0-	-0-	-100			B13Z	Specimen dissolved
	272		1.2555	-0-	-0-	-100			B13Z	Specimen dissolved
Polypropylene	32	10.66	0.8828	0.8850	0.0022	.25	67*	72*	B18X	Discolored to dark reddish-brown
	32	10.38	0.8493	0.8514	0.0021	.25		72*	B18X	Discolored to dark reddish-brown
	91	9.87	0.8184	0.8191	0.0007	.086		67*	B18Y	Dark reddish-brown
	91	11.56	0.9651	0.9655	0.0004	.041		70*	B18Y	Dark reddish-brown
	273	11.50	0.9128	0.9163	0.0035	.38		>100*	B18Z	Black - some apparent surface attack
	273	8.66	0.7220	0.7248	0.0028	.39		>100*	B18Z	Black - some apparent surface attack
Dry Powder TFE	32		0.0006	0.0012	0.0006	100			B19X	No apparent reaction
(MS 122)	91		0.0015	0.0022	0.0007	46.7			B19Y	No apparent reaction
	273		0.0011	0.0009	-0.0002	-18.2			B19Z	No apparent reaction

*Shore "D" Hardness Values.

TABLE 2.2-12

DATA INDICATIVE OF THE COMPATIBILITY OF GASEOUS NITROGEN TRIFLUORIDE AT 344 K (160 F) AND 3.45 MN/m^2 (500 PSIA) WITH ELASTOMERIC MATERIALS

Material	Exposure Time, days	Specimen Surface Area, cm^2	Specimen Weights, gm			Tensile Strength, psi		Modulus at 100% Elongation, psi		Ultimate Elongation, %		Wallace Hardness		Test No.	Observations
			Initial	Final	Change	Initial	Final	Initial	Final	Initial	Final	Initial	Final		
Kalrez	32	8.73	1.5268	1.5445	0.0177	1778 ± 119	2194	694 ± 38	935	173 ± 17	174	68 ± 0.5	69	B15X	Color changed to white
	32	8.65	1.5327	1.5505	0.0178		1980		932		194		69	B15X	Color changed to white
	91	8.96	1.5295	1.5331	0.0059		2064		786		174		70	B15Y	No apparent reaction
	91	8.95	1.5272	1.5307	0.0035		1840		831		165		69	B15Y	No apparent reaction
	274	8.33	1.5314	1.6739	0.1425		1470		838		191		55	B15Z	Swollen, elongated and stuck together
	274	8.81	1.5357	1.6769	0.1424		1205		933		139		44	B15Z	Swollen, elongated and stuck together
Viton, Class I	33	8.72	1.2276	1.2313	0.0037	1394 ± 44	344	453 ± 23		230 ± 16	87	74 ± 0.5	70	B17X	No apparent reaction
	33	8.79	1.2318	1.2352	0.0034		348				96		70	B17X	No apparent reaction
	91	8.25	1.2303	1.2374	0.0076		1337		488		235		73	B17Y	No apparent reaction
	91	8.73	1.2160	1.2233	0.0073		1225		485		218		73	B17Y	No apparent reaction
	268	8.69	1.2339		0.0301		**		**		**		**	B17Z	Elongated and swollen, stuck
	268	8.63	1.2325		0.0308		**		**		**		**	B17Z	to other "O"rings.
Silastic LS-53	31	8.53	0.9509	0.9475	-0.0034	661 ± 124	239	361 ± 13		159 ± 25	70	63 ± 0.6	69	B16X	No apparent reaction
	31	8.48	0.9517	0.9471	-0.0046		104				35		68	B16X	No apparent reaction
	91	8.60	0.9512	0.9534	0.0022		631		441		148		68	B16Y	No aparrent reaction
	91	8.66	0.9574	0.9587	0.0013		235				52		69	B16Y	No apparent reaction
	276	8.53	0.9535	0.9359	-0.0176		**		**		**		53	B16Z	Specimen cracked and soft to touch
	276	8.60	0.9543	0.9348	-0.0195		**		**		**		46	B16Z	Specimen cracked and soft to touch

**Specimens deteriorated to the stage that valid measurements were not possible.

after 270 days of exposure, and (2) the Kalrez exhibited some decrease in tensile strength and hardness which was not prohibitive, but the surface was visibly affected by the exposure. The test specimens were heated to 422 K (300 F) for two days during the 270 day exposure period due to an oven controller problem, and this event may have accelerated some deterioration of the materials. Photographs of the Kalrez and Silastic LS 53 after the 270 day exposure period are shown in Figure 2.2.17.

The data obtained from the exposure of Viton, Class II to nitrogen trifluoride at 344 K (160 F) and at pressures ranging from 3.45 MN/m^2 (500 psia) to 17.24 MN/m^2 (2500 psia) are presented in Table 2.2-13. The significant items to note from the data are as follows: (1) short-term exposure of Viton, Class II for 90 days or less at pressures below 10.34 MN/m^2 (1500 psia) is possible without significant degradation in properties and (2) longer-term exposure, between 90 and 270 days, results in the unacceptable degradation of use-properties at pressures ranging from 3.45 MN/m^2 (500 psia) to 17.24 MN/m^2 (2500 psia). Photographs of the Vitons exposed to gaseous nitrogen trifluoride for 270 days are shown in Figure 2.2.18.

The chemical analyses of the nitrogen trifluoride recovered from the static exposure of the non-metallic materials are given in Table 2.2-14. Generally, at 195 K (-78 C), no effect of materials on the decomposition of nitrogen trifluoride is observed except that approximately one percent decomposition of nitrogen trifluoride is observed in the presence of Silastic LS 53, and a few percent decomposition in the presence of Krytox after the sample composition is corrected for air contamination.

At 344 K (160 F), the Fluorosilicone FS 3451 caused a 17 wt % decrease in the nitrogen trifluoride assay; changes in nitrogen trifluoride content ranging from 1 to 3 weight percent were observed in the presence of Kalrez, Silastic LS 53, polypropylene, Carbon CJPS, Carbon CDJ-83, Kevlar, Krytox, and dry powder PTFE (MS-22). The remaining materials, Vitons, polytetrafluoroethylene, FEP Teflon, PFA Teflon, Rulon and Kel-F 81, had a negligible effect on the decomposition of nitrogen trifluoride.

TABLE 2.2-13

DATA INDICATIVE OF THE COMPATIBILITY OF GASEOUS NITROGEN TRIFLUORIDE AT 344 K (160 F) AND PRESSURES RANGING FROM 3.45 TO 17.24 MN/m^2 (500 TO 2500 PSIA) WITH VITON, CLASS II

Exposure Time, days	Pressure		Specimen Surface Area, cm^2	Specimen Weights, gm			Tensile Strength, psi		Modulus at 100% Elongation, psi		Ultimate Elongation, %		Wallace Hardness		Test No.	Observations
	MN/m^2	psia		Initial	Final	Change	Initial	Final	Initial	Final	Initial	Final	Initial	Final		
33	3.45	500	9.23	1.2291	1.3034	0.0043	1483	1419	992	983	167	174	92 ± 0.8	93	B17X	Slightly swollen
33	3.45	500	9.04	1.2824	1.2865	0.0041	± 107	1364	± 20	957	± 14	157		92	B17X	Slightly swollen
91	3.45	500	9.10	1.3053	1.3129	0.0076		1340		1021		148		91	B17Y	No apparent reaction
91	3.45	500	9.17	1.2999	1.3075	0.0076		1344		978		157		91	B17Y	No apparent reaction
266	3.45	500	9.02	1.2905	1.3590	0.0685		**		**		**		37	B17Z	Stuck to other "O"rings
268	3.45	500	9.02	1.2694	1.3301	0.0607		712		460		279		38	B17Z	Stuck to other "O"rings
33	8.62	1250	9.11	1.2879	1.2974	0.0095		1370		928		188		92	C17X	Swollen and elongated
33	8.62	1250	9.11	1.2879	1.2979	0.0100		1314		928		173		92	C17X	Swollen and elongated
33	13.44	1950	8.98	1.2916	1.3054	0.0138		643		-0-		61		91	D17X	Swollen and elongated
33	13.44	1950	9.04	1.2882	1.3018	0.0136		894		894		100		91	D17X	Swollen and elongated
29	17.24	2500	9.04	1.2615	1.2707	0.0092		1448		885		166		80	*DNX	Swollen and elongated
29	17.24	2500	9.04	1.2521	1.2613	0.0092		1293		841		192		80	*DNX	Swollen and elongated
96	10.34	1500	9.04	1.2565	1.2690	0.0125		1390		799		227		70	C17Y	Swollen and elongated
96	10.34	1500	9.10	1.2646	1.2777	0.0131		1343		837		200		77	C17Y	Swollen and elongated
96	17.24	2500	8.97	1.2615	1.2751	0.0136		936		870		113		82	D17Y	Swollen and elongated
96	17.24	2500	9.16	1.2722	1.2843	0.0121		816		769		113		72	D17Y	Swollen and elongated
269	10.34	1500	9.03	1.2902	1.3625	0.0723		**		**		**		38	C17Z	Swollen and elongated
269	10.34	1500	9.00	1.2790	1.3488	0.0698		**		**		**		40	C17Z	Swollen and elongated
269	17.24	2500	9.04	1.2770	1.3154	0.0384		**		**		**		**	D17Z	Cracked, elongated
269	17.24	2500	9.04	1.2887	1.3331	0.0444		**		**		**		**	D17Z	deformed, sticky and soft to the touch.

**Specimens deteriorated to the stage that valid measurements were not possible

TABLE 2.2-14

CHEMICAL COMPOSITION OF NITROGEN TRIFLUORIDE RECOVERED FROM STATIC EXPOSURE TESTS WITH NON-METALLIC MATERIALS

Test No.	Type of Exposure	Composition, Weight Percent							Cylinder of Origin for the NF_3	Table No. in Which Specimen Data are Reported
		NF_3	Active Fluorides as HF	N_2	O_2/CO	CF_4	CO_2	N_2O		
A8X	Liquid/Vapor NF_3, 195 K, 33 days All Teflons	99.16	0.0004	0.35	0.44	0.0078	0.0012	0.051	H81136	2.2-6
A8Y	Liquid/Vapor NF_3, 195 K, 90 days All Teflons	98.49	<.0002	0.84	0.59	0.0070	0.013	0.061	H81136	2.2-6
A8Z	Liquid/Vapor NF_3, 195 K, 273 days All Teflons		0.0056	No Chromatographic Analysis					17319-C	2.2-6
B8X	3.45 MN/m^2 NF_3, 344 K, 30 days All Teflons	97.91	0.20	0.85	0.15	0.66	0.14	0.082	17228-C	2.2-10
B8Y	3.45 MN/m^2 NF_3, 344 K, 90 days All Teflons	98.91	<.0002	0.55	0.41	0.0070	0.048	0.072	H81136	2.2-10
B8Z	3.45 MN/m^2 NF_3, 344 K, 272 days All Teflons	98.57	0.086	0.47	0.15	0.46	0.18	0.078	17319-C	2.2-10
C8X	8.62 MN/m^2 NF_3, 344 K, 30 days Polytetrafluoroethylene	97.29	0.42	1.20	0.32	0.65	0.055	0.056	17228-C	2.2-10
C8Y	10.34 MN/m^2 NF_3, 344 K, 90 days Polytetrafluoroethylene	98.41	0.063	0.22	0.27	0.97	0.011	0.049	H55957	2.2-10
C8Z	10.34 MN/m^2 NF_3, 344 K, 273 days Polytetrafluoroethylene	97.86	0.11	0.69	0.33	0.97	0.0036	0.030	H55957	2.2-10
D8X	13.44 MN/m^2 NF_3, 344 K, 30 days Polytetrafluoroethylene	98.07	0.45	0.71	Tr	0.66	0.041	0.070	17228-C	2.2-10
*DNX	17.24 MN/m^2 NF_3, 344 K, 29 days Polytetrafluoroethylene, Kel F and Viton Class II	99.12	0.031	0.098	0.21	0.44	0.059	0.037	H55957 P178684	2.2-10 2.2-11 2.2-12
D8Y	17.24 MN/m^2 NF_3, 344 K, 89 days Polytetrafluoroethylene	98.61	0.036	0.11	0.19	1.00	0.0088	0.043	H55957	2.2-10
D8Z	17.24 MN/m^2 NF_3, 344 K, 269 days Polytetrafluoroethylene	98.40	0.084	0.27	0.22	1.00	0.0036	0.027	H55957	2.2-10
	Cylinder 17228-C	98.68	0.17	0.20	0.10	0.75	0.013	0.083		
	Cylinder 17319-C	98.72	0.10	0.13	0.45	0.51	0.016	0.070		
	Cylinder H55957	98.68	0.0002	0	0.24	1.03	0.011	0.048		
	Cylinder H81136	99.56	0.0001	0	0.35	0.009	0	0.074		
	Cylinder P178684	99.68	0.0003	0	0.29	0.017	0	0.014		

TABLE 2.2-14 (cont.)

Test No.	Type of Exposure	Composition, Weight Percent: NF_3	Active Fluorides as HF	N_2	O_2/CO	CF_4	CO_2	N_2O	Cylinder of Origin for the NF_3	Table No. in Which Specimen Data are Reported
A9X	Liquid/Vapor NF_3, 195 K, 34 days Kel-F 81	98.67	0.025	0.21	0.42	0.59	0.033	0.058	17319-C	2.2-6
A9Y	Liquid/Vapor NF_3, 195 K, 90 days Kel-F 81	99.11	<.0002	0.34	0.46	0.0068	0.024	0.062	H81136	2.2-6
A9Z	Liquid/Vapor NF_3, 195 K, 272 days Kel-F 81	98.42	0.11	0.35	0.60	0.46	0.024	0.042	17319-C	2.2-6
B9X	3.45 MN/m^2 NF_3, 344 K, 32 days Kel-F 81	98.36	0.029	0.66	0.28	0.47	0.11	0.083	17319-C	2.2-11
B9Y	3.45 MN/m^2 NF_3, 344 K, 90 days Kel-F 81	98.89	0.0011	0.62	0.40	0.0069	0.015	0.068	H81136	2.2-11
B9Z	3.45 MN/m^2 NF_3, 344 K, 272 days Kel-F 81	98.79	0.076	0.53	Tr.	0.46	0.055	0.031	17319-C	2.2-11
C9X	8.62 MN/m^2 NF_3, 344 K, 30 days Kel-F 81	97.71	0.46	0.89	0.17	0.66	0.056	0.053	17228-C	2.2-11
C9Y	10.34 MN/m^2 NF_3, 344 K, 91 days Kel-F 81	98.05	0.074	0.51	0.35	0.97	0.005	0.035	H55957	2.2-11
C9Z	10.34 MN/m^2 NF_3, 344 K, 273 days Kel-F 81	97.87	0.10	0.69	0.33	0.97	0.005	0.038	H55957	2.2-11
D9X	13.44 MN/m^2 NF_3, 344 K, 30 days Kel-F 81	98.02	0.44	0.63	0.14	0.66	0.055	0.065	17228-C	2.2-11
D9Y	17.24 MN/m^2 NF_3, 344 K, 88 days Kel-F 81	98.43	0.0058	0.25	0.25	1.00	0.022	0.045	H55957	2.2-11
D9Z	17.24 MN/m^2 NF_3, 344 K, 269 days Kel-F 81	98.58	0.069	0.10	0.21	1.00	0.015	0.026	H55957	2.2-11
A10X	Liquid/Vapor NF_3, 195 K, 31 days Carbon CDJ-83	98.37	0.13	0.23	0.40	0.79	0.015	0.066	17228-C	2.2-6
A10Y	Liquid/Vapor NF_3, 195 K, 92 days Carbon CDJ-83	99.41	0.0004	0.20	0.32	0.014	0	0.048	P178684	2.2-6
A10Z	Liquid/Vapor NF_3, 195 K, 273 days Carbon CDJ-83	98.72	0.080	0.34	0.35	0.44	0.023	0.043	17319-C	2.2-6
B10X	3.45 MN/m^2, 344 K, 32 days Carbon CDJ-83	97.64	0.019	0.97	0.49	0.48	0.31	0.088	17319-C	2.2-8

TABLE 2.2-14 (cont.)

Test No.	Type of Exposure	Composition, Weight Percent: NF_3	Active Fluorides as HF	N_2	O_2/CO	CF_4	CO_2	N_2O	Cylinder of Origin for the NF_3	Table No. in Which Specimen Data are Reported
A13Z	Liquid/Vapor NF_3, 195 K, 273 days Fluorosilicone FS 3451	98.61	0.023	0.37	0.40	0.49	0.020	0.091	17319-C	2.2-6
B13X	3.45 MN/m^2 NF_3, 344 K, 30 days Fluorosilicone FS 3451	97.48	0.15	0.84	Tr	0.67	0.78	0.084	17228-C	2.2-8
B13Z	3.45 MN/m^2, 344 K, 272 days Fluorosilicone FS 3451	81.72	7.19	4.28	1.86	0.68	2.48	1.26	17319-C	2.2-8
A14X	Liquid/Vapor NF_3, 195 K, 35 days Kevlar	98.37	0.0042	0.44	0.56	0.56	0.015	0.049	17319-C	2.2-6
A14Y	Liquid/Vapor NF_3, 195 K, 93 days Kevlar	98.43	0.006	0.39	0.43	0.61	0.023	0.10	17319-C	2.2-6
A14Z	Liquid/Vapor NF_3, 195 K, 275 days Kevlar	98.64	0.013	0.34	0.38	0.54	0.0072	0.090	17319-C	2.2-6
B14X	3.45 MN/m^2 NF_3, 344 K, 32 days Kevlar	No Data							17319-C	2.2-8
B14Y	3.45 MN/m^2 NF_3, 344 K, 92 days Kevlar	96.71	0.001	2.01	0.48	0.47	0.23	0.099	17319-C	2.2-8
B14Z	3.45 MN/m^2 NF_3, 344 K, 274 days Kevlar	97.72	0.10	1.27	Tr.	0.46	0.34	0.11	17319-C	2.2-8
A15X	Liquid/Vapor NF_3, 195 K, 33 days Kalrez	98.83	0.0052	0.15	0.33	0.60	0.019	0.059	17319-C	2.2-7
A15Y	Liquid/Vapor NF_3, 195 K, 91 days Kalrez	99.45	0.0002	0.21	0.30	0.014	0	0.027	P178684	2.2-7
A15Z	Liquid/Vapor NF_3, 195 K, 274 days Kalrez	98.78	0.066	0.25	0.29	0.50	0.036	0.074	17319-C	2.2-7
B15X	3.45 MN/m^2 NF_3, 344 K, 32 days Kalrez	97.56	0.013	0.96	0.36	0.48	0.55	0.074	17319-C	2.2-9
B15Y	3.45 MN/m^2 NF_3, 344 K, 91 days Kalrez (Corrected for air)	88.60 97.01	0.0036 0.0039	9.22 2.47	2.04 0.33	0.012 0.013	0.11 0.12	0.016 0.018	P178684	2.2-9
B15Z	3.45 MN/m^2 NF_3, 344 K, 274 days Kalrez	95.03	0.068	2.70	0	0.54	1.15	0.51	17319-C	2.2-9
A16X	Liquid/Vapor NF_3, 195 K, 33 days Silastic LS-53	98.75	0.004	0.17	0.38	0.62	0.020	0.053	17319-C	2.2-7
A16Y	Liquid/Vapor NF_3, 195 K, 91 days Silastic LS-53	99.34	0.0002	0.20	0.41	0.014	0	0.047	P178684	2.2-7
A16Z	Liquid/Vapor NF_3, 195 K, 270 days Silastic LS-53	97.95	0.011	0.93	0.54	0.47	0.018	0.080	17319-C	2.2-7

TABLE 2.2-14 (cont.)

Test No.	Type of Exposure	Composition, Weight Percent: NF_3	Active Fluorides as HF	N_2	O_2/CO	CF_4	CO_2	N_2O	Cylinder of Origin for the NF_3	Table No. in Which Specimen Data are Reported
B10Y	3.45 MN/m^2, 344 K, 90 days Carbon CDJ-83	99.45	0.0084	0.24	0.18	0.014	0.065	0.041	P178684	2.2-8
B10Z	3.45 MN/m^2, 344 K, 273 days Carbon CDJ-83	95.96	0.087	2.68	0	0.49	0.52	0.27	17319-C	2.2-8
A11X	Liquid/Vapor NF_3, 195 K, 34 days Carbon CJPS	98.76	0.024	0.15	0.36	0.60	0.040	^.064	17319-C	2.2-6
A11Y	Liquid/Vapor NF_3, 195 K, 92 days Carbon CJPS	99.21	<.0002	0.31	0.43	0.014	0	0.028	P178684	2.2-6
A11Z	Liquid/Vapor NF_3, 195 K, 270 days Carbon CJPS	98.52	0.031	0.40	0.50	0.45	0.028	0.061	17319-C	2.2-6
B11X	3.45 MN/m^2 NF_3, 344 K, 32 days Carbon CJPS	95.74	0.0083	2.36	1.23	0.47	0.12	0.082	17319-C	2.2-8
B11Y	3.45 MN/m^2 NF_3, 344 K, 91 days Carbon CJPS	98.89	0.023	0.24	0.18	0.014	0.065	0.041	P178684	2.2-8
B11Z	3.45 MN/m^2 NF_3, 344 K, 273 days Carbon CJPS	95.33	0.10	2.75	1.15	0.44	0.12	0.11	17319-C	2.2-8
A12X	Liquid/Vapor NF_3, 195 K, 33 days Krytox	98.65	0.019	0.20	0.42	0.61	0.031	0.068	17319-C	2.2-6
A12Z	Liquid/Vapor NF_3, 195 K, 273 days Krytox	91.12	0.028	6.56	1.78	0.41	0.041	0.057	17319-C	2.2-6
B12X	3.45 MN/m^2 NF_3, 344 K, 32 days Krytox	97.19	0.099	1.61	0.52	0.48	0.032	0.066	17319-C	2.2-8
B12Z	3.45 MN/m^2 NF_3, 344 K, 272 days Krytox	98.42	0.072	0.95	Tr.	0.43	0.068	0.064	17319-C	2.2-8
A12*X	Liquid/Vapor NF_3, 195 K, 32 days Vacuum-stripped Krytox	98.77	0.0016	0.17	0.25	0.75	0.013	0.043	H55957 P178684	2.2-6
B12*X	3.45 MN/m^2 NF_3, 344 K, 31 days Vacuum-stripped Krytox	99.02	0.0010	0.071	0.074	0.75	0.036	0.0010	H55957 P178684	2.2-8
A13X	Liquid/Vapor NF_3, 195 K, 33 days Fluorosilicone FS 3451	99.55	0.0020	0.088	0.30	0.016	0.0076	0.032	P178684	2.2-6
A13Y	Liquid/Vapor NF_3, 195 K, 91 days Fluorosilicone FS 3451	99.33	0.0006	0.25	0.35	0.015	0	0.057	P178684	2.2-6

TABLE 2.2-14 (cont.)

Test No.	Type of Exposure	Composition, Weight Percent: NF_3	Active Fluorides as HF	N_2	O_2/CO	CF_4	CO_2	N_2O	Cylinder of Origin for the NF_3	Table No. in Which Specimen Data are Reported
B16X	3.45 MN/m^2 NF_3, 344 K, 31 days Silastic LS-53	96.82	0.038	1.06	0.90	0.47	0.65	0.065	17319-C	2.2-9
B16Y	3.45 MN/m^2 NF_3, 344 K, 91 days Silastic LS-53	99.44	0.0034	0.25	0.15	0.014	0.11	0.027	P178684	2.2-9
B16Z	3.45 MN/m^2 NF_3, 344 K, 276 days Silastic LS-53	97.88	0.063	1.12	Tr.	0.48	0.37	0.081	17319-C	2.2-9
A17X	Liquid/Vapor NF_3, 195 K, 33 days Vitons	99.46	Tr.	0.11	0.36	0.008	0.0029	0.060	H81136	2.2-7
A17Y	Liquid/Vapor NF_3, 195 K, 91 days Vitons	99.32	0.0002	0.26	0.37	0.015	0	0.032	P178684	2.2-7
A17Z	Liquid/Vapor NF_3, 195 K, 33 days Vitons	98.50	0.13	0.36	0.16	0.73	0.019	0.097	17228-C	2.2-7
B17X	3.45 MN/m^2 NF_3, 344 K, 33 days Vitons	99.35	Tr.	0.25	0.18	0.0073	0.15	0.073	H81136	2.2-9 2.2-12
B17Y	3.45 MN/m^2 NF_3, 344 K, 91 days Vitons	99.14	0.0002	0.42	0.17	0.014	0.23	0.031	P178684	2.2-9 2.2-12
B17Z	3.45 MN/m^2 NF_3, 344 K, 268 days Vitons	93.43	1.16	0.021	1.38	0.70	2.40	0.91	17228-C	2.2-9 2.2-12
C17X	8.62 MN/m^2 NF_3, 344 K, 33 days Viton, Class II	99.24	0.0002	0.23	0.37	0.0074	0.070	0.072	H81136	2.2-12
C17Y	10.34 MN/m^2 NF_3, 344 K, 94 days Viton, Class II	99.07	0.0005	0.058	0.029	0.68	0.11	0.045	H55957 P178684	2.2-12
C17Z	10.34 MN/m^2 NF_3, 344 K, 269 days Viton Class II	98.43	0.030	0.22	0.22	0.99	0.084	0.026	H55957	2.2-12
D17X	13.44 MN/m^2 NF_3, 344 K, 33 days Viton, Class II	99.38	0.0005	0.15	0.35	0.0075	0.061	0.058	H81136	2.2-12
D17Y	17.24 MN/m^2 NF_3, 344 K, 96 days Viton, Class II	No data							H55957 P178684	2.2-12
D17Z	17.24 MN/m^2 NF_3, 344 K, 269 days Viton, Class II	98.55	0.036	0.15	0.15	0.98	0.11	0.03	H55957	2.2-12
B18X	3.45 MN/m^2 NF_3, 344 K, 32 days Polypropylene	97.49	0.027	1.24	0.34	0.47	0.35	0.074	17319-C	2.2-8

TABLE 2.2-14 (cont.)

Test No.	Type of Exposure	Composition, Weight Percent: NF_3	Active Fluorides as HF	N_2	O_2/CO	CF_4	CO_2	N_2O	Cylinder of Origin for the NF_3	Table No. in Which Specimen Data are Reported
B18Y	3.45 MN/m^2 NF_3, 344 K, 91 days Polypropylene	99.44	0.0036	0.25	0.10	0.014	0.15	0.040	P178684	2.2-8
B18Z	3.45 MN/m^2 NF_3, 344 K, 273 days Polypropylene	97.31	0.11	1.75	Tr.	0.43	0.31	0.085	17319-C	2.2-8
B19X	3.45 MN/m^2 NF_3, 344 K, 32 days Dry Powder TFE (MS 122)	97.85	0.062	1.07	0.40	0.47	0.073	0.072	17319-C	2.2-8
B19Y	3.45 MN/m^2 NF_3, 344 K, 91 days Dry Powder TFE (MS 122)	97.92	0.0029	1.47	0.50	0.015	0.047	0.046	P178684	2.2-8
B19Z	3.45 MN/m^2 NF_3, 344 K, 273 days Dry Powder TFE (MS 122)	97.42	0.12	1.85	Tr.	0.45	0.061	0.094	17319-C	2.2-8

2.0, Experiment Results and Discussion (cont.)

2.3 FRACTURE MECHANICS/TOUGHNESS TESTS

The purpose of the fracture mechanics/toughness tests is to determine the susceptibility of the selected metals to the phenomenon of stress-crack corrosion. The resistance of a structure to failure by flaw propagation under a rising load is a mechanical property known as the fracture toughness, K_{IC}, for plane strain loading conditions. To predict the behavior of structures the fracture toughness of the material of interest is determined by testing fatigue precracked specimens and determining the stress intensity, K, (a quantity which depends on the crack length, load, and specimen geometry) at which the specimen fails by rapid or unstable crack propagation. This critical value of stress intensity is defined as K_{IC}. Tests for K_{IC} determination are conducted in air under conditions specified by ASTM-E399-74.

Enhanced flaw propagation is also observed under constant load conditions below the critical stress intensity level in specific environment-material combinations and is referred to as stress corrosion cracking (SCC). In testing in environments such as salt water or nitrogen trifluoride a stress intensity less than K_{IC} is imposed and the precrack in the specimen grows in a slow stable fashion as a result of environmental interactions. If the jaws of the test fixture remain fixed (constant deflection), the stress intensity, K, will drop from $K<K_{IC}$ to $K = K_{ISCC}$ as a result of the crack growth. K_{ISCC} is the limiting or critical stress intensity below which no crack growth will occur for a defined material-environment combination under sustained load conditions. Some materials have poor stress corrosion cracking resistance and experience a major decrease in K when exposed to service environments; others do not. Nine candidate metals were selected for evaluation. The candidates with their heat-treat conditions are itemized in Table 2.3-1.

2.3.1 Test Procedures

Plate was procured for all the specimens except Inconel 718 and Arde 301. One inch plate was procured for the titanium alloys, CRES 347 and C-1018 steel materials. 3/4 inch plate was procured for aluminum, CRES 17-4PH and 250 Maraging materials. Inconel 718 specimens were machined from 3 inch diameter bar stock and the Arde' 301 specimens were machined from 1/16 inch thick sheet.

The program was conducted in two steps: (1) the fracture toughness measurements were made initially in an air environment and (2) the stress corrosion cracking tests which were subsequently conducted in various nitrogen trifluoride environments. With the exception of the Arde 301, one welded and one parent specimen of each material were prepared as compact

TABLE 2.3-1

MATERIALS SELECTED FOR THE NITROGEN TRIFLUORIDE STRESS CORROSION CRACKING TESTING WITH HEAT TREATMENT AND WELD FILLER WIRE

Material As-Received	GMA Welded Weld Filler Metal	Heat Treatment Condition Tested	Hardness After Heat Treatment
Al 2219-T37	Al 2319	Weld and Parent were aged at 340-360°F for 18 hrs, air cooled, (MIL-H-6088). Brings to T87 condition.	R_B 77
CRES 347	--	Received in annealed condition.	--
CRES 347	CRES 349	Stress relieved at 1600-1700°F for 2 hrs, air cooled.	R_B 88
CRES 17-4 PH H 1025	CRES 17-4 PH	Weld and parent were solution treated at 1875-1925°F for 30 min, air cooled below 90°F and aged at 1025°F for 4 hrs (MIL-H-6875).	R_C 37
Inconel 718	Inconel 718	Weld and parent were solution treated at 1925-1975°F for 1 hr, water quenched, precipitation treated at 1385-1415°F for 10 hrs, furnace cooled to 1185-1215°F and held there until a total precipitation time of 20 hrs had been reached, air cooled.	--
Ti 5Al-2.5 Sn ELI	--	Received in annealed condition.	--
Ti 5Al-2.5 Sn ELI	Ti 5Al-2.5 Sn ELI	Stress relieved at 985-1015°F for 4 hrs, air cooled.	R_C 31
Ti 6Al-4V	--	Received in the STA condition.	--
Ti 6Al-4V	Ti 6Al-4V	Stress relieved at 1000°F for 4 hrs, air cooled.	R_C 41
C-1018 Steel	--	Annealed	--
C-1018 Steel	Linde 85	Stress relieved 1 hr at 1085-1115°F for 1 hr, air cooled.	R_B 81
VM-250 Maraging Steel	250 Maraging Steel	Aged at 900°F for 3 hrs	--
Arde 301	--	As-received welded-sheet.	

tension specimens in the configuration shown in Figure 2.3.1. The welded specimens were double vee grooved and GMA welded with the filler metals identified in Table 2.3-1. These specimens were subjected to fracture toughness testing in air.

The metal specimens used in the stress corrosion cracking tests in the nitrogen trifluoride environments were bolt-loaded and had the same weld preparation and crack orientation as the compact tension specimens. A photograph of the typical stress corrosion cracking specimen is shown in Figure 2.3.2. The crack plane orientation with respect to the rolling direction of the test specimens is given in Table 2.3-2. The rolling direction code given in the table is pictorially presented in Figure 2.3.3. Because the Arde 301 was available only in the form of a thin sheet, it was tested for stress corrosion cracking effect only in the configuration shown in Figure 2.3.4.

The fracture toughness tests, were conducted first to determine the preload value for the bolt-loaded constant stress corrosion cracking specimens. Specimens were precracked and tested per ASTM E399-74 by Atlas Testing Laboratories.* At the end of each test the resulting K value was judged either valid or invalid according to the criteria in ASTM E399-74. Values judged invalid were still useful for estimating the loading required for the stress corrosion cracking specimens. The fracture toughness values obtained are reported in Table 2.3-3.

The bolt-load constant deflection test specimens were supplied by Atlas Testing Laboratories in the machined and precracked condition. In addition, each of the bolt loaded specimens was loaded at Atlas Testing Laboratories to a value which corresponded to K_i, see Table 2.3-3, as determined by the following equation:

$$P = \frac{0.8\, K_i\, B\, W^{1/2}}{f\left(\frac{a}{W}\right)} \qquad (1)$$

where:

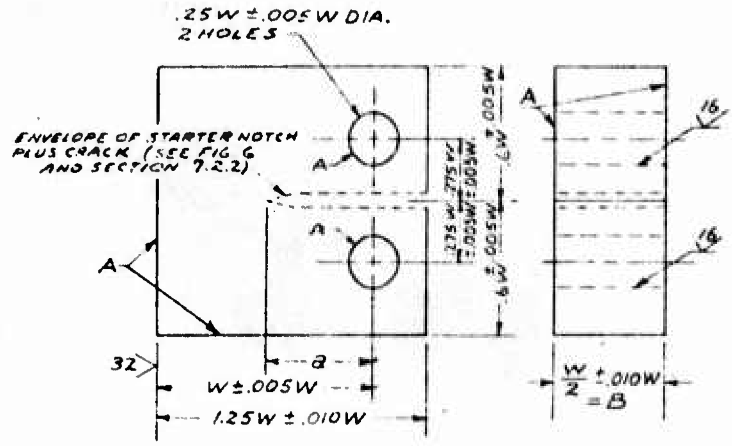
P is the bolt load on the specimen; in.-lb

K_i is 80 percent of the stress intensity which caused failure in the fracture toughness tests

B is specimen thickness in in.

w is specimen width in in.

*Atlas Testing Laboratories, Los Angeles, CA.



NOTE 1—A surfaces shall be perpendicular and parallel as applicable to within 0.002 W TIR.

NOTE 2—The points of intersection of the crack starter tips with the two specimen faces shall be equally distant from either pin hole center to within 0.005 W.

NOTE 3—Integral or attachable knife edges for clip gage attachment to the crack mouth may be used (see Fig. 7 and 7.5.2)

Metric Equivalents

in.	0.002	0.005	0.010
mm	0.05	0.13	0.25

Figure 2.3.1. Compact Tension Specimen - Standard Proportions and Tolerances

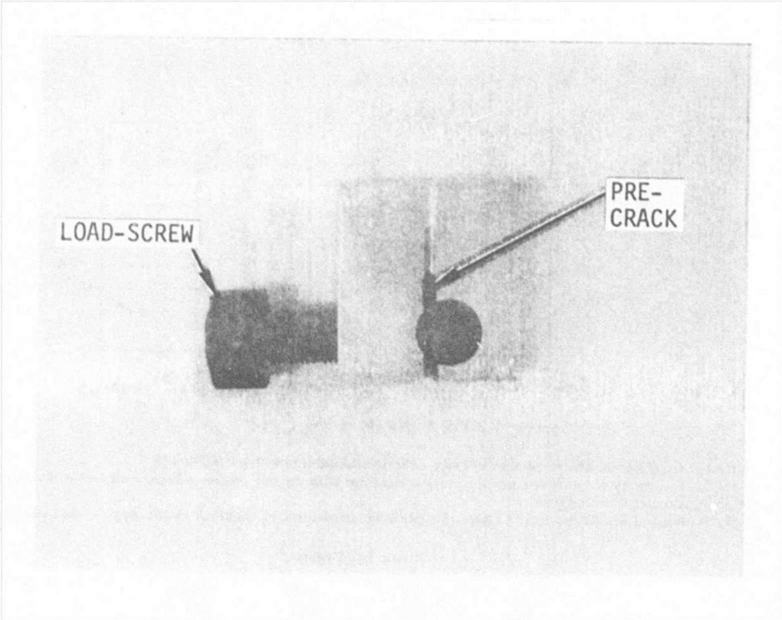


Figure 2.3.2. Photograph of a Bolt-Loaded Stress Corrosion Cracking Specimen

TABLE 2.3-2

SPECIMEN MATERIAL AND CRACK PLANE ORIENTATION FOR SPECIMENS

Material	Crack Plane Orientation With Respect to Rolling Direction
Al 2219-T87	T-L
Welded	In Weld
CRES 347	T-L
Welded	In Weld
17-4 PH - H1025	T-L
Welded	In Weld
Inconel 718	T-S/S-T
Welded	In Weld
Ti 5Al-2.5 Sn ELI	T-L
Welded	In Weld
Ti 6Al-4V STA	T-L
1018 Steel	T-L
Welded	In Weld
250 Maraging Steel	
Welded	In Weld
Arde 301	--

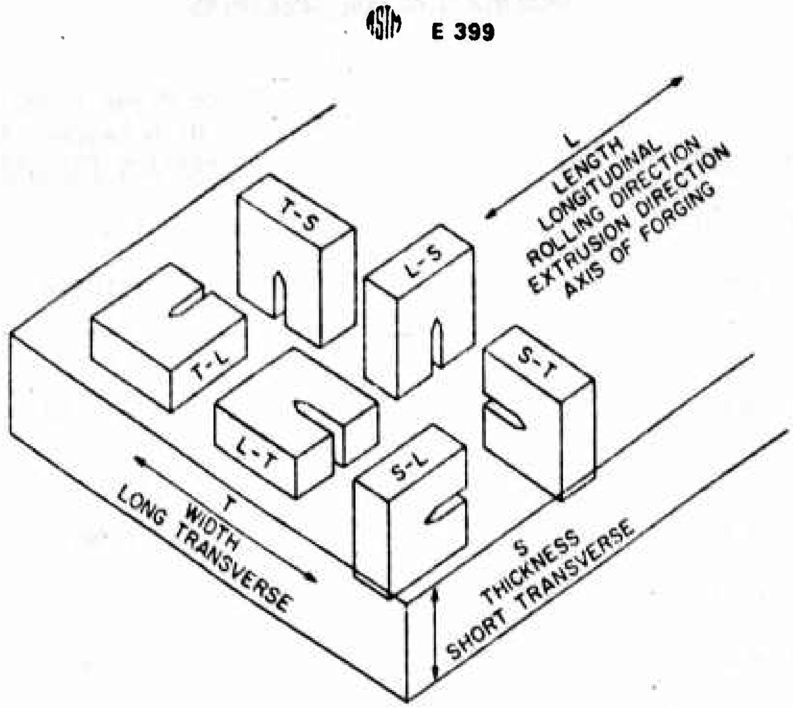
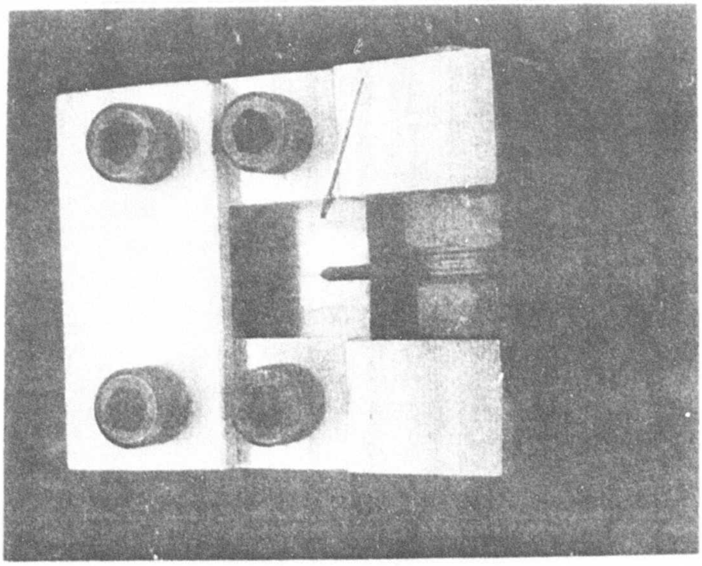


Figure 2.3.3. Specimen and Pre-Crack Plane Orientation with Respect to Material Rolling Direction



MAG. 3X

Figure 2.3.4. Photograph of the Arde 301 Qualitative Crack Growth Specimen. Sheet Specimen is Indicated by the Arrow

TABLE 2.3-3

FRACTURE TOUGHNESS VALUES FOR THE CANDIDATE MATERIALS

Material		K_{Iq} lbf x $10^{-3} \cdot$ in.$^{-3/2}$	$K_i = .8\ K_{Iq}$ lbf x $10^{-3} \cdot$ in.$^{-3/2}$	Valid K_{IC}
Al 2219 T87	Parent	24.2	19.4	Yes
	Welded	16.1	12.9	No
CRES 347	Parent	45.2	36.2	No
	Welded	42.8	34.2	No
CRES 17-4 PH	Parent	64.0	51.2	Yes
	Welded	77.6	62.1	No
Inconel 718	Parent	81.6	65.3	Yes
	Welded	74.9	59.9	No
Ti 5Al-2.5 Sn	Parent	63.9	51.1	Yes
	Welded	82.9	66.3	No
Ti 6Al-4V	Parent	36.1	28.9	No
	Welded	60.9	48.7	No
C-1018 Steel	Parent	62.2	49.8	Yes
	Welded	51.1	40.9	No
250 Maraging Steel	Welded	76.4	61.1	Yes
Arde 301	Welded	--	--	--

2.3, Fracture Mechanics/Toughness Tests (cont.)

a is crack length in in.,

and $f(\frac{a}{w})$ is a function determined by specimen geometry.

The relative displacement was measured across the crack mouth using a clip gage and was recorded for each specimen.

At ALRC the specimens were cleaned with isopropanol, bolt-loaded to the clip gage displacement determined at Atlas Testing Laboratories, and placed in test bombs. Specimens were exposed to each of the three nitrogen trifluoride test environments: 2500 psia gas, at 344 K (160 F), 500 psia gas at 344 K (160 F) and liquid at 195 K.

At the end of 30 and 90 days specimens were removed, the cracks were measured to the nearest 0.1 mm on both sides of each specimen by means of a microscope with calibrated stage, and the specimens were re-exposed to the environment. After 180 days of total exposure the specimens were removed from the bombs and unloaded by loosening the bolt with the clip gage in place. Once the clip gage displacement had been noted the specimen was placed in an Instron tensile test machine, loaded, and the load necessary to open the specimen crack mouth to the bolt-loaded clip-gage displacement was recorded as P_{ISCC}. Following the determination of P_{ISCC} each specimen was broken open in air, the P_q determined and the crack front measured at its quarter points per ASTM-E399-74. The K_{ISCC} and K_q were then calculated for each specimen by:

$$K_{ISCC} = \frac{P_{ISCC}\, f(\frac{a}{w})}{B\, W^{1/2}} \qquad (2a)$$

and

$$K_q = \frac{P_q\, f(\frac{a}{w})}{B\, w^{1/2}} \qquad (2b)$$

where:

K_{ISCC} is the limiting or critical stress intensity below which no crack growth will occur for a defined material-environment combination under sustained load;

P_{ISCC} is the residual load left in the specimen after the 180 days of exposure; K_q is the stress intensity where specimens failed by rapid or unstable crack propagation;

P_q is the load taken from the load-clip gage displacement curve as defined by ASTM-E399-74;

and B, w, $f(\frac{a}{w})$ are as defined for equation (1).

2.3, Fracture Mechanics/Toughness Tests (cont.)

2.3.2 Experimental Results

The specimen data from the stress corrosion cracking tests after 180 days exposure are presented in Table 2.3-4. The data have been subdivided according to the nitrogen trifluoride environment and the parent or welded condition of the test specimens. In Table 2.3-5 the average K_{ISCC} values are compared with the K_i values generated by the fracture toughness testing. The K_{Iq} values from the fracture toughness tests are compared with the K_q values in Table 2.3-6.

Typical fracture surfaces of the specimens exposed to nitrogen trifluoride are shown in Figure 2.3.5 through Figure 2.3.12. In some cases, the bands on the fracture surfaces correspond to the 30, 90, and 180 day exposure periods as the specimens were removed from the nitrogen trifluoride, examined, and re-exposed. Weld defects such as poor penetration and gas porosity can be seen on many welded specimen fracture surfaces.

The comparison of the average K_{ISCC} for the materials in Table 2.3-5 shows some interesting material-environment interactions. In the 160°F, 500 psia gaseous environment welded Al 2219 did not show evidence of stress corrosion cracking whereas the parent material did crack (see Figure 2.3.5). Parent and welded CRES 17-4PH behaved similarly in the 160°F, 500 psia environment (see Figure 2.3.7a and 2.3.7b). The Ti 6Al-4V parent specimen was severely cracked (see Figure 2.3-10a). No evidence of stress corrosion cracking was found in either the parent or welded CRES 347, Figure 2.3.6, nor in Inconel 718, Figure 2.3.8.

In the 2500 psia environment welded Inconel 718 specimens did not stress corrosion crack nor did welded C-1018 steel specimens. In contrast both parent and welded Ti 6Al-4V specimens cracked to such an extent that they were removed from the environment after 30 days of exposure. The welded Ti 6Al-4V specimens in the 160°F, 2500 psia nitrogen trifluoride cracked all the way (see Figure 2.3.10b), thus indicating a K_{ISCC} value much lower than that listed for the parent material in Table 2.3-5. Other materials that cracked in the 160°F 2500 psia NF_3 environment were parent C-1018 and welded 250 Maraging steels, see Figure 2.3.11a and 2.3.12.

Parent Ti 5Al-2.5 Sn was the only material to exhibit stress corrosion cracking in liquid nitrogen trifluoride at 108°F, see Figure 2.3.9. No indication of stress corrosion cracking was seen on the fracture surfaces of the other specimens tested in liquid nitrogen trifluoride.

TABLE 2.3-4

DATA OBTAINED FROM THE SPECIMENS AFTER 180 DAYS EXPOSURE IN NITROGEN TRIFLUORIDE FOR STRESS CORROSION CRACKING EVALUATION

Material	NF_3 Environment	Specimen No.	a in.	w in.	B in.	$\frac{a}{w}$	$f(\frac{a}{w})$	P_{ISCC} lb	K_{ISCC} $lbf x 10^{-3} \cdot in.^{-3/2}$	P_q lb.	K_q $lbf x 10^{-3} \cdot in.^{-3/2}$	K_i $lbf x 10^{-3} \cdot in.^{-3/2}$
Al 2219-T87 Parent	160°F, 500 psia	1-1	0.942	1.502	0.671	0.627	15.155	910	16.77	1410	25.99	19.4
		1-2	0.870	1.501	0.666	0.580	12.517	1205	18.49	1745	26.77	
		1-3	0.876	1.503	0.666	0.583	12.670	1195	18.54	1748	27.12	
Al 2219-T87 Welded	160°F, 500 psia	1-6w	0.729	1.505	0.668	0.484	9.174	1030	11.53	1476	16.52	12.9
		1-7w	0.822	1.504	0.668	0.547	11.129	970	13.18	1325	18.00	
		1-8w	0.746	1.505	0.671	0.496	9.481	655	7.54*	1510	17.39	
Al 2219-T87 Welded	-108°F, liquid	1-4w	0.769	1.504	0.669	0.511	9.937	875	10.60	1425	17.26	12.9
		1-5w	0.745	1.506	0.667	0.495	9.453	927	10.71	1400	16.17	
		1-9w	0.746	1.503	0.672	0.495	9.500	1000	11.53	1498	17.27	
CRES 347 Parent	160°F, 500 psia	2-1	1.026	2.003	0.910	0.512	9.965	4200	32.50	6700	51.84	36.2
		2-2	1.020	2.016	0.910	0.506	9.777	4280	32.38	6860	51.91	
		2-3	0.999	2.004	0.910	0.499	9.561	4990	37.03	7160	53.14	
CRES 347 Welded	160°F, 500 psia	2-4w	1.036	2.003	0.910	0.517	10.121	4460	35.05	6320	49.66	34.2
		2-5w	0.984	2.005	0.910	0.491	9.346	4820	34.96	7000	50.77	
CRES 17-4PH Parent	160°F, 500 psia	3-1	1.237	1.508	0.665	0.820	42.182	535	27.64	920	47.52	51.2
		3-2	1.247	1.505	0.664	0.829	44.309	545	29.65	810	44.06	
		3-3	1.300	1.509	0.664	0.861	53.934	362	23.94	565	37.36	
CRES 17-4PH Welded	160°F, 500 psia	3-5w	0.765	1.505	0.666	0.508	9.847	5090	61.34	9180	110.63	62.1
		3-9w	0.772	1.507	0.666	0.512	9.967	5600	68.27	8000	97.53	
		3-12w	0.770	1.506	0.666	0.511	9.937	5290	64.31	8980	109.18	
CRES 17-4PH Welded	160°F, 2500 psia	3-6w	1.212	1.502	0.665	0.807	38.98	890	42.56	1260	60.26	62.1
		3-10w	0.828	1.504	0.664	0.551	11.281	4540	62.90	7450	103.21	
		3-11w	1.256	1.506	0.665	0.834	45.76	709	39.76	1370	76.83	
CRES 17-4PH Welded	-108°F, liquid	3-4w	0.754	1.511	0.665	0.499	9.575	4040	47.32	8760	102.61	62.1
		3-7w	0.756	1.516	0.663	0.499	9.566	4470	52.38	9120	106.87	
		3-8w	0.778	1.512	0.665	0.515	10.037	4420	54.25	8560	105.07	
Inconel 718 Parent	160°F, 500 psia	4-1	0.988	1.998	0.913	0.494	9.448	9450	69.18	14950	109.45	65.3
		4-2	1.029	1.998	0.918	0.515	10.051	9590	74.29	11050	85.60	
		4-3	0.997	1.992	0.895	0.501	9.618	6250	47.59	13800	105.07	
Inconel 718 Welded	160°F, 500 psia	4-6w	0.911	2.004	0.910	0.455	8.442	8690	56.95	13100	85.85	59.9
		4-9w	0.985	2.000	0.915	0.492	9.393	8810	63.95	10000	72.59	
		4-11w	1.048	1.996	0.912	0.525	10.373	7640	61.51	8700	70.04	
Inconel 718 Welded	160°F, 2500 psia	4-7w	0.951	2.000	0.904	0.475	8.945	8610	60.24	12500	87.46	59.9
		4-10w	0.979	2.005	0.925	0.488	9.278	8740	61.91	11850	83.94	
		4-12w	0.974	2.002	0.908	0.487	9.231	8790	63.16	11250	80.83	
Inconel 718 Welded	-108°F, liquid	4-5w	0.953	1.994	0.912	0.478	9.007	9220	57.49	10100	70.64	59.9
		4-8w	0.991	2.005	0.930	0.494	9.442	9410	67.47	11100	79.59	
Ti 5Al-2.5Sn Parent ELI	-108°F, liquid	5-1	1.608	2.010	0.914	0.800	37.421	1365	39.42	No P_q, K_q		51.1
		5-2	1.608	2.006	0.911	0.837	46.726	1130	40.92	could be		
		5-3	1.501	2.006	0.910	0.748	27.796	1830	39.47	determined		
Ti 5Al 2.5Sn Welded ELI	-108°F, liquid	5-4w	1.016	2.006	0.910	0.506	9.792	8500	64.58	11750	89.27	66.3
		5-5w	0.970	2.006	0.910	0.484	9.152	9400	66.75	12400	88.05	

TABLE 2.3-4 (cont.)

Material	NF_3 Environment	Specimen No.	a in.	w in.	B in.	$\frac{a}{w}$	$f(\frac{a}{w})$	P_{ISCC} lb	K_{ISCC} $lbf x 10^{-3} \cdot in.^{-3/2}$	P_q lb.	K_q $lbf x 10^{-3} \cdot in.^{-3/2}$	K_i $lbf x 10^{-3} \cdot in.^{-3/2}$
Ti 6Al-4V Parent	160°F, 500 psia	6-1	1.730	2.004	0.913	0.863	54.510	455	19.19	364	15.31	28.9
		6-2	1.747	2.013	0.911	0.868	56.027	450	19.51	425	18.42	
		6-3	1.713	2.009	0.912	0.853	51.159	467	18.48	500	19.79	
		6-5	1.788	2.002	0.910	0.893	65.151	325	16.44	320	16.19	
		6-6	1.754	2.008	0.916	0.874	57.951	347	15.49	330	14.73	
Ti 6Al-4V Parent	160°F,2500 psia	6-4	1.736	2.007	0.910	0.865	55.076	375	16.04	365	15.61	28.9
Ti 6Al-4V Welded	160°F,2500 psia	6-7w 6-8w	These specimens cracked all the way through after 30 days. K_{ISCC} must be less than that for the parent specimen.									
AISI 1018 Parent	160°F,2500 psia	7-1	1.214	2.002	0.925	0.606	13.893	4500	47.78	4390	46.60	49.8
		7-2	0.936	2.004	0.920	0.467	8.736	7560	50.71	8890	59.63	
		7-3	0.956	2.004	0.918	0.477	8.984	7400	51.16	7550	52.19	
AISI 1018 Welded	160°F,2500 psia	7-4w	0.989	2.003	0.918	0.494	9.428	6160	44.70	8230	59.72	40.9
		7-5w	0.971	2.009	0.920	0.483	9.146	6200	43.49	7800	54.71	
250 Marage Welded	160°F,2500 psia	8-1w	0.831	1.506	0.672	0.552	-	3820	55.23	6710	97.01	61.1
		8-2w	1.030	1.513	0.665	0.681	19.439	3220	76.52	4120	97.91	
		8-3w	0.805	1.507	0.654	0.532	10.682	4610	61.34	7080	94.20	

TABLE 2.3-5

COMPARISON OF $K_i = 0.8\ K_{Iq}$ WITH AVERAGE K_{ISCC} VALUES

Material		$K_i = 0.8\ K_{Iq}$ lbf x $10^{-3} \cdot in.^{-3/2}$	K_{ISCC} Values for the Various NF_3 Test Environments: 2500 psia gas 160°F lbf x $10^{-3} \cdot in.^{3/2}$	500 psia gas 160°F lbf x $10^{-3} \cdot in.^{-3/2}$	Liquid -108°F lbf x $10^{-3} \cdot in.^{-3/2}$
Al-2219-T87	Parent	19.4		17.93	
	Welded	12.9		12.35*	10.94*
CRES 347	Parent	36.2		33.97*	
	Welded	34.2		35.00*	
CRES 17-4 PH	Parent	51.2		27.07	
	Welded	62.1	41.16+	64.64*	51.32*
Inconel 718	Parent	65.3		63.68*	
	Welded	59.9	61.77*	60.80*	62.48*
Ti 5Al-2.5 Sn	Parent	51.1			39.94
	Welded	66.3			65.66*
Ti 6Al-4V	Parent	28.9	16.04	17.82	
	Welded	48.7	Cracked Through		
C-1018 Steel	Parent	49.8	47.77**		
	Welded	40.9	44.09*		
250 Maraging Steel	Welded	61.1	64.36		

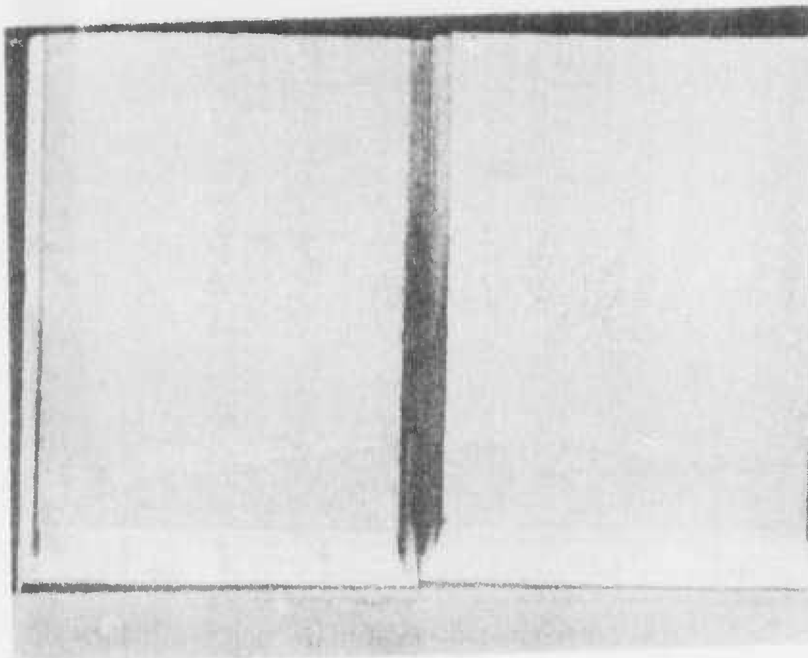
*No crack growth occurred.
**Only one of three specimens showed cracking and its value was used.
+Only two of three specimens showed cracking and their values were averaged.

TABLE 2.3-6

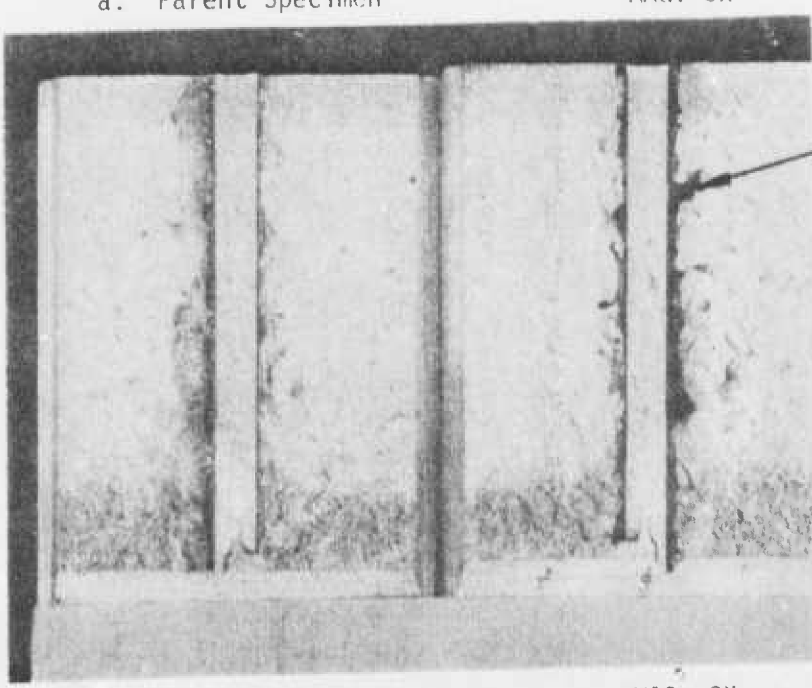
COMPARISON OF FRACTURE TOUGHNESS K_{Iq} AND STRESS CORROSION CRACKING K_q VALUES

Material		K_{Iq} lbf x $10^{-3}\cdot$in.$^{-3/2}$	K_q After Exposure to NF_3 Environment: 160°F, 2500 psi gas lbf x $10^{-3}\cdot$in.$^{-3/2}$	160°F, 500 psi gas lbf x $10^{-3}\cdot$in.$^{-3/2}$	-108°F, liquid lbf x $10^{-3}\cdot$in.$^{-3/2}$
Al-2219-T87	Parent	24.2		26.61	
	Welded	16.1		17.30	16.90
CRES 347	Parent	45.2		52.30	
	Welded	42.8		50.22	
CRES 17-4 PH	Parent	64.0		42.98	
	Welded	77.6	68.54	105.78	104.85
Inconel 718	Parent	81.6		100.03	
	Welded	74.9	84.08	76.16	75.11
Ti 5Al-2.5 Sn	Parent	63.9			*
	Welded	82.9			88.66
Ti 6Al-4V	Parent	36.1	*	*	
	Welded	60.9	*		
C-1018 Steel	Parent	62.2	55.91		
	Welded	51.1	57.21		
250 Maraging Steel	Welded	76.4	96.37		

*A P_q value could not be determined, therefore no K_q is listed.



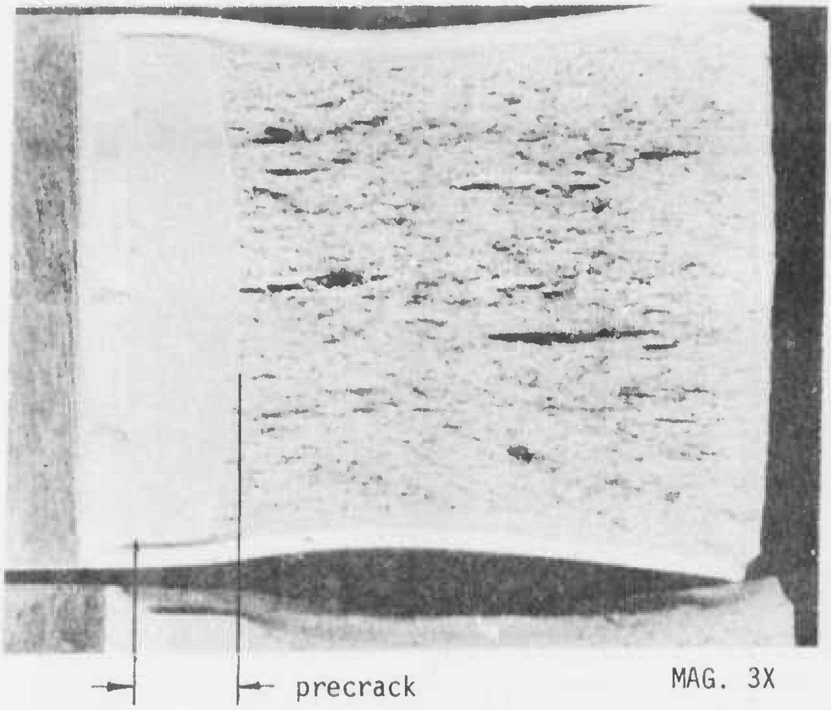
a. Parent Specimen MAG. 3X



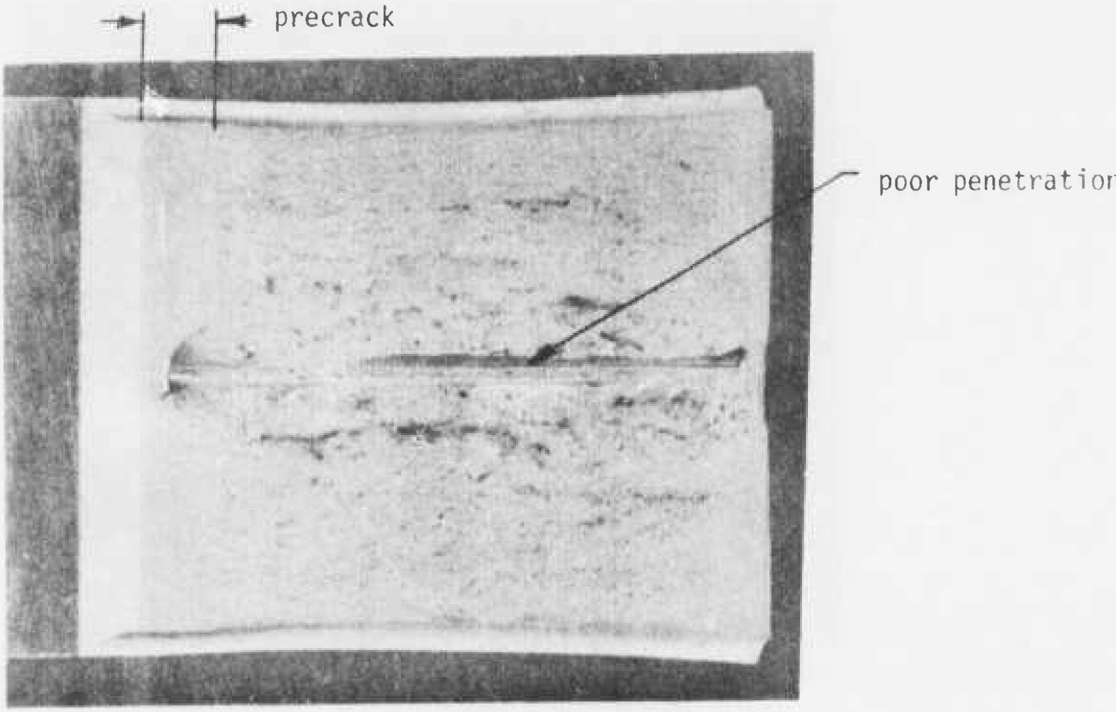
MAG. 3X

b. Welded Specimen

Figure 2.3.5. Al 2219-T87 K_{ISCC} Specimens After Exposure To 500 psia Gaseous NF_3 at 160°F

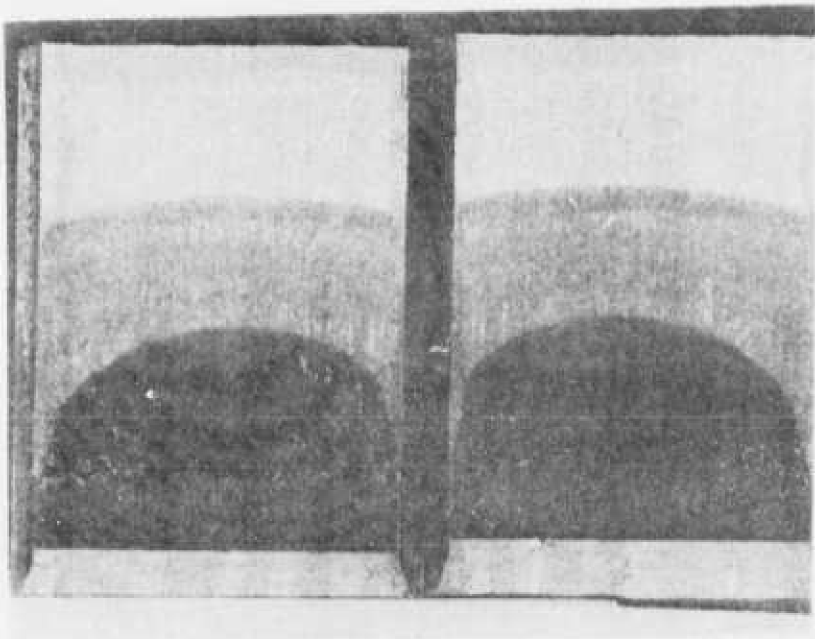


a. Parent Specimen

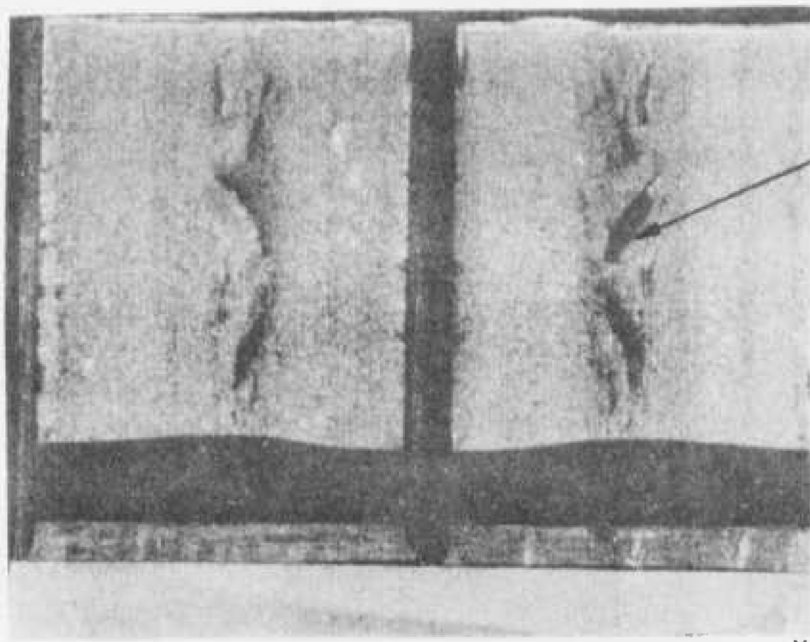


b. Welded Specimen

Figure 2.3.6. CRES 347 Exposed to 500 psia Gaseous NF_3 at 160°F

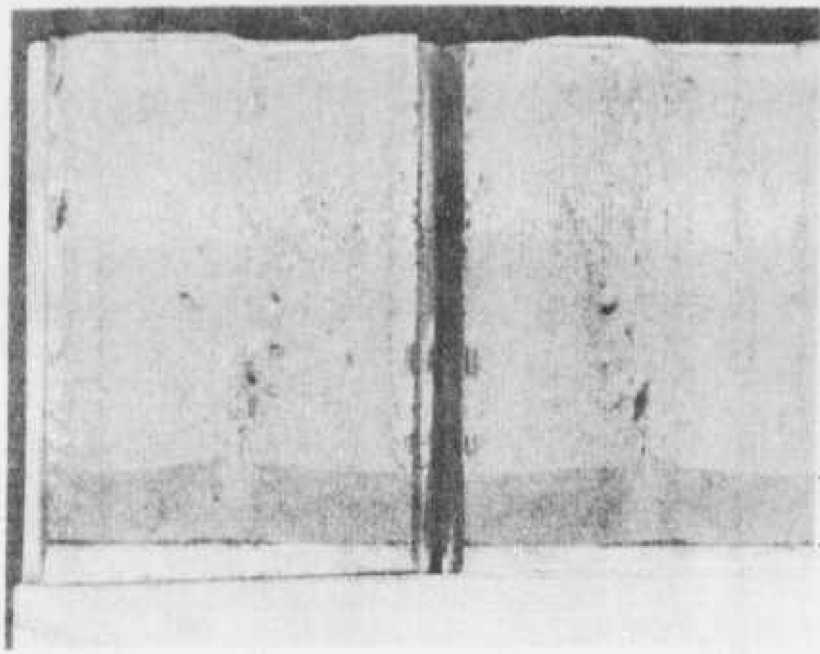


a. Parent Specimen Exposed to Gaseous NF_3 at 500 psia and 160°F

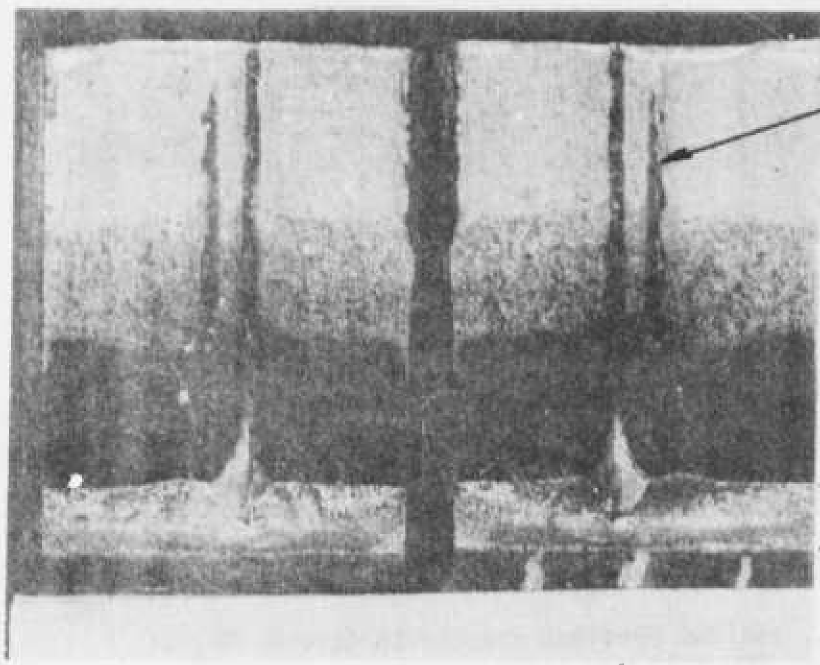


b. Welded Specimen Exposed to Gaseous NF_3 at 500 psia and 160°F

Figure 2.3.7. CRES 17-4 PH Exposed to Liquid and Gaseous NF_3

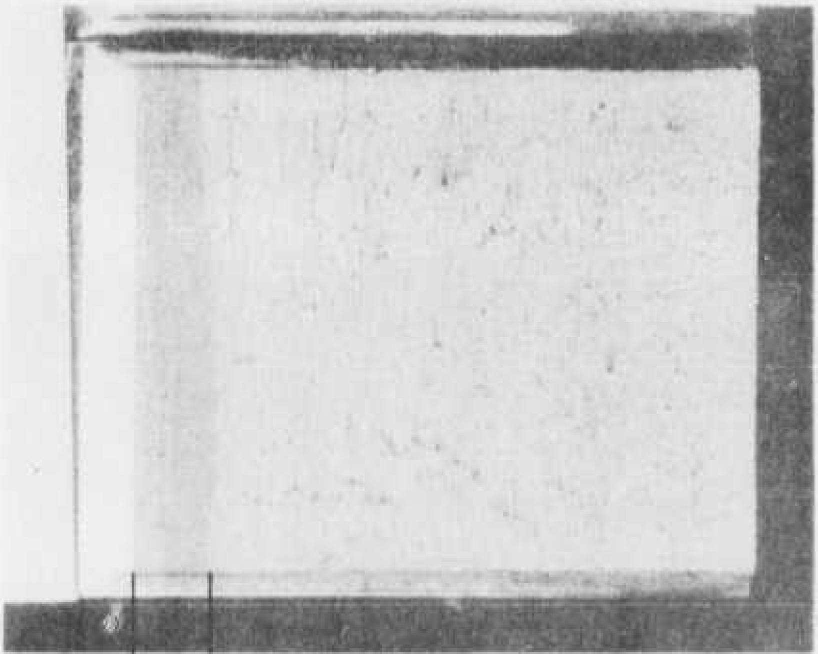


c. Welded Specimen Exposed to Liquid NF_3 at -78°C

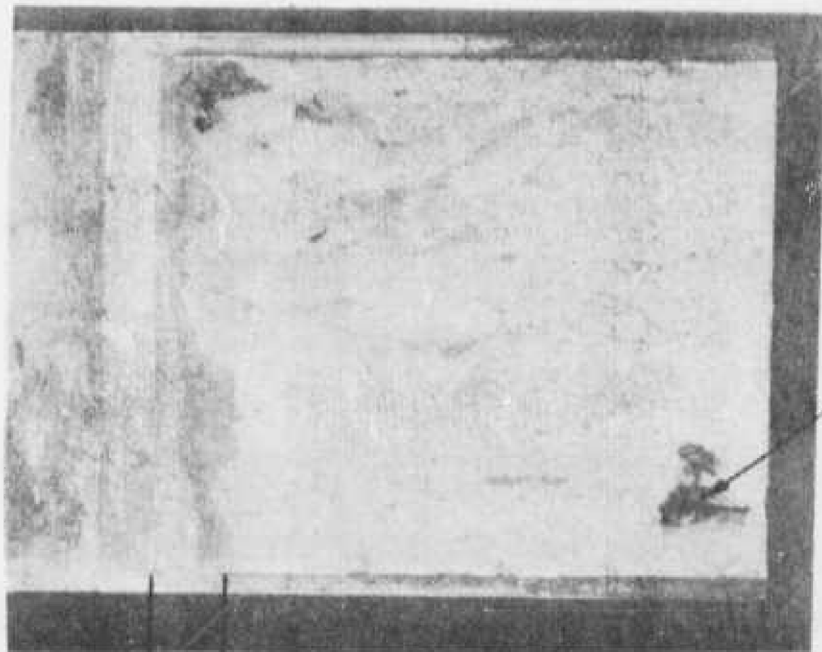


d. Welded Specimen Exposed to Gaseous NF_3 at 2500 psia and 160°F

Figure 2.3.7. CRES 17-4 PH Exposed to Liquid and Gaseous NF_3 (cont.)

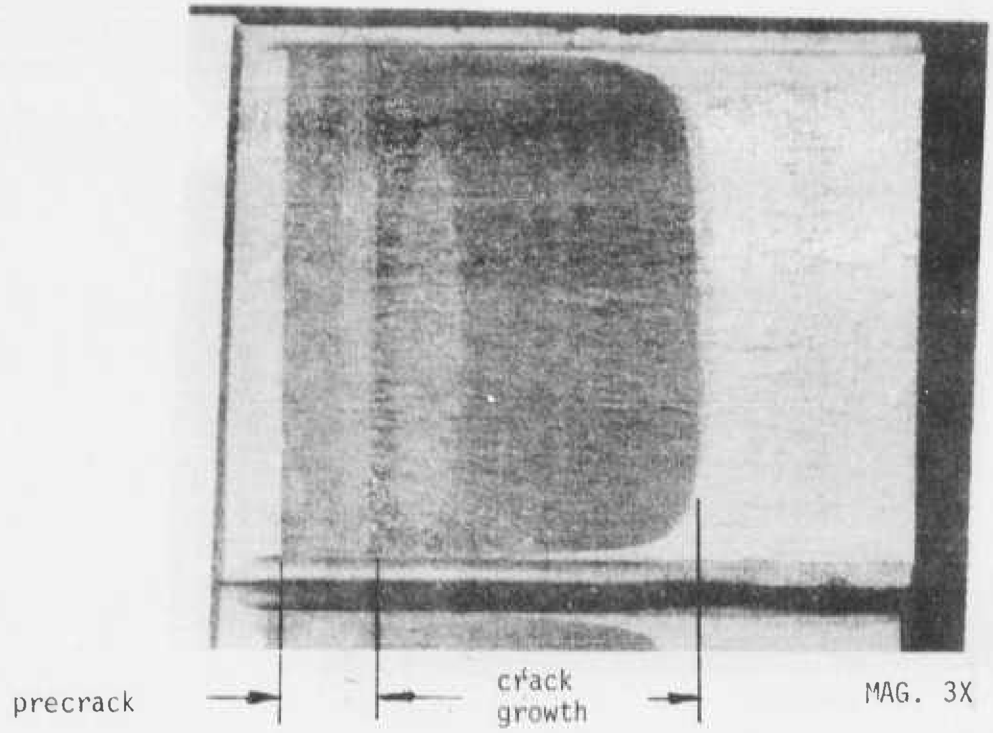


a. Parent Specimen Exposed to Gaseous NF_3 at 500 psia and 160°F

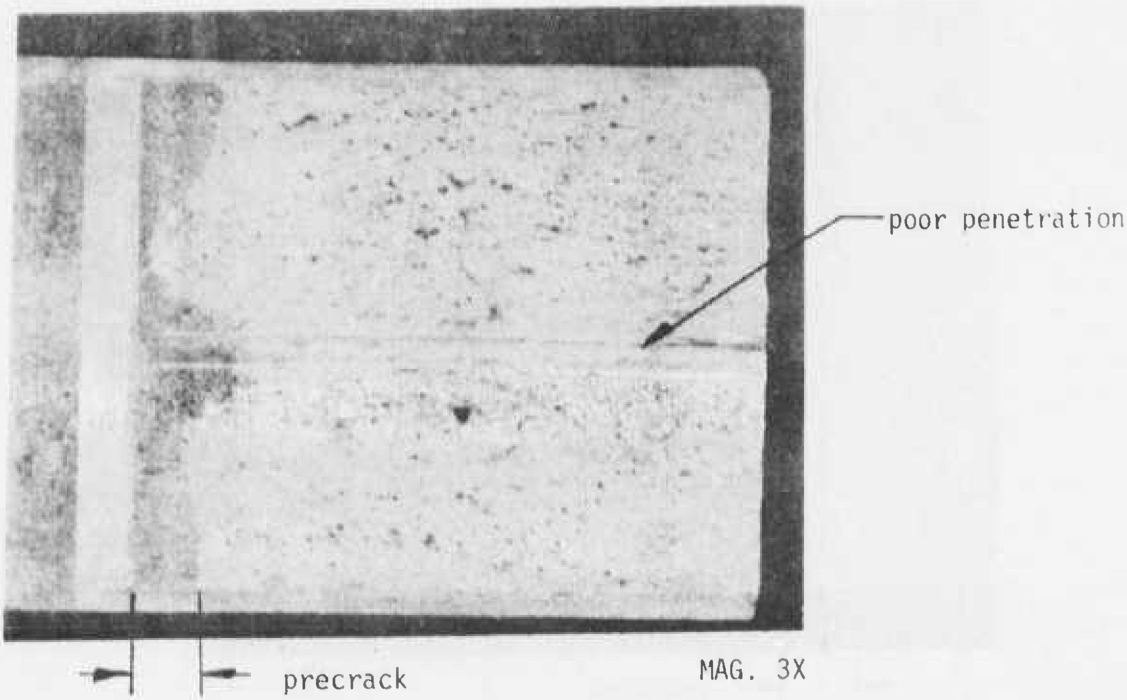


b. Welded Specimen Exposed to Liquid NF_3 at -78°C

Figure 2.3.8. Inconel 718 Specimens Exposed to Liquid and Gaseous NF_3

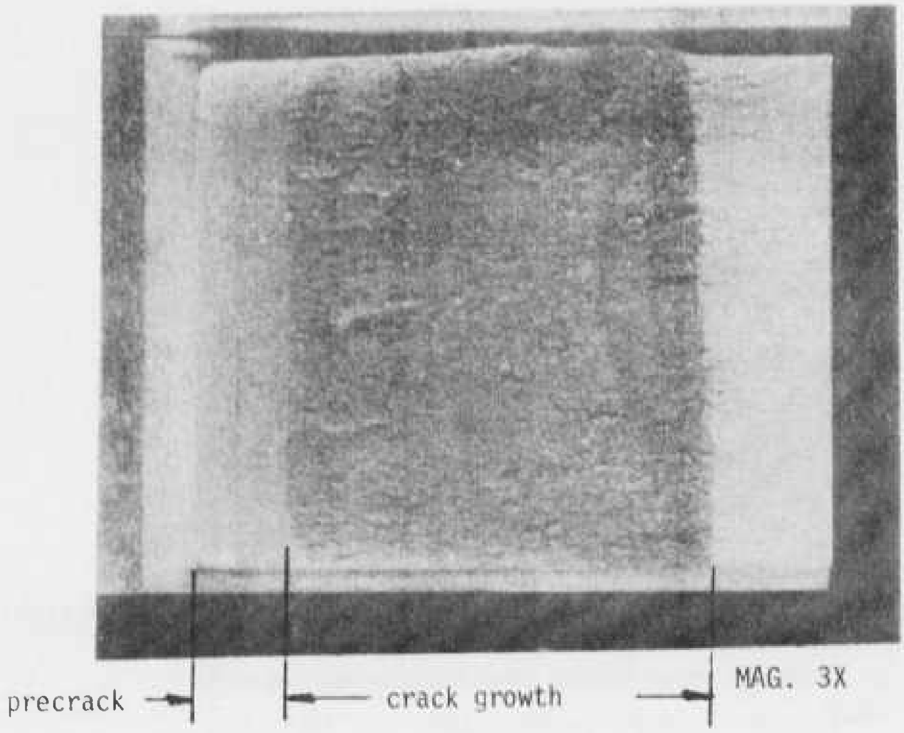


a. Parent Specimen

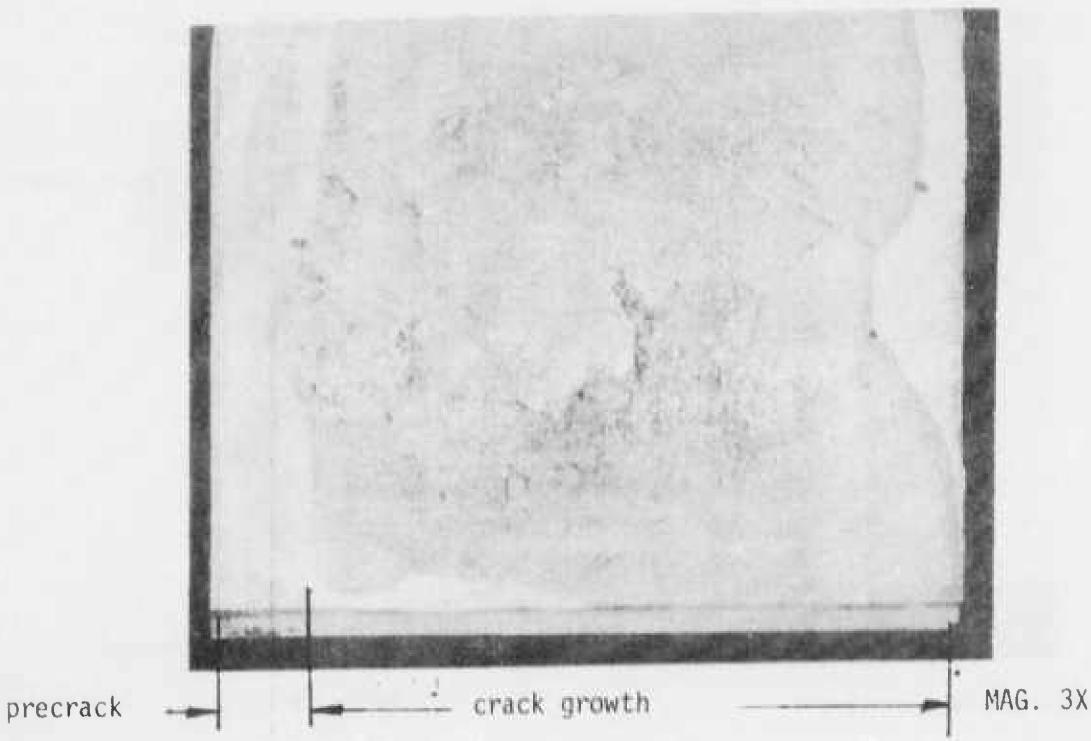


b. Welded Specimen

Figure 2.3.9. Ti 5Al-2.5 Sn ELI Specimens Exposed to Liquid NF_3 at -78°C

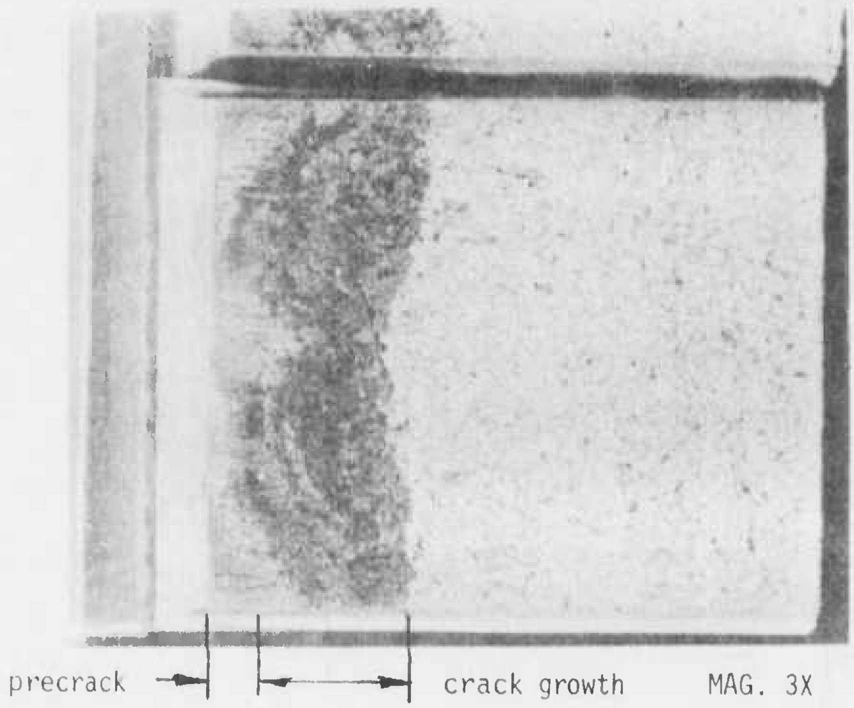


a. Welded Specimen Exposed to 2500 psia Gaseous NF_3

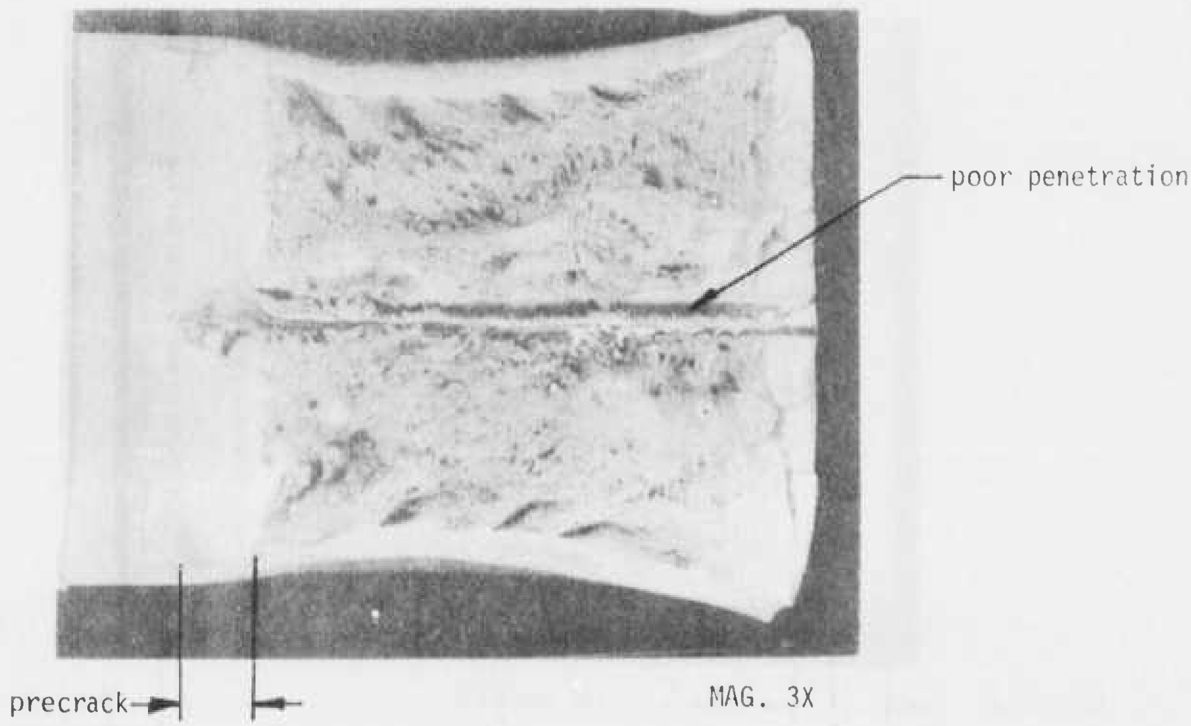


b. Welded Specimen Exposed to 2500 psia Gaseous NF_3

Figure 2.3.10. Ti 6Al-4V in a Gaseous NF_3 Environment at 160°F



a. Parent Specimen



b. Welded Specimen

Figure 2.3.11. C-1018 Steel in a 2500 psia Gaseous NF_3 Environment at 160°F

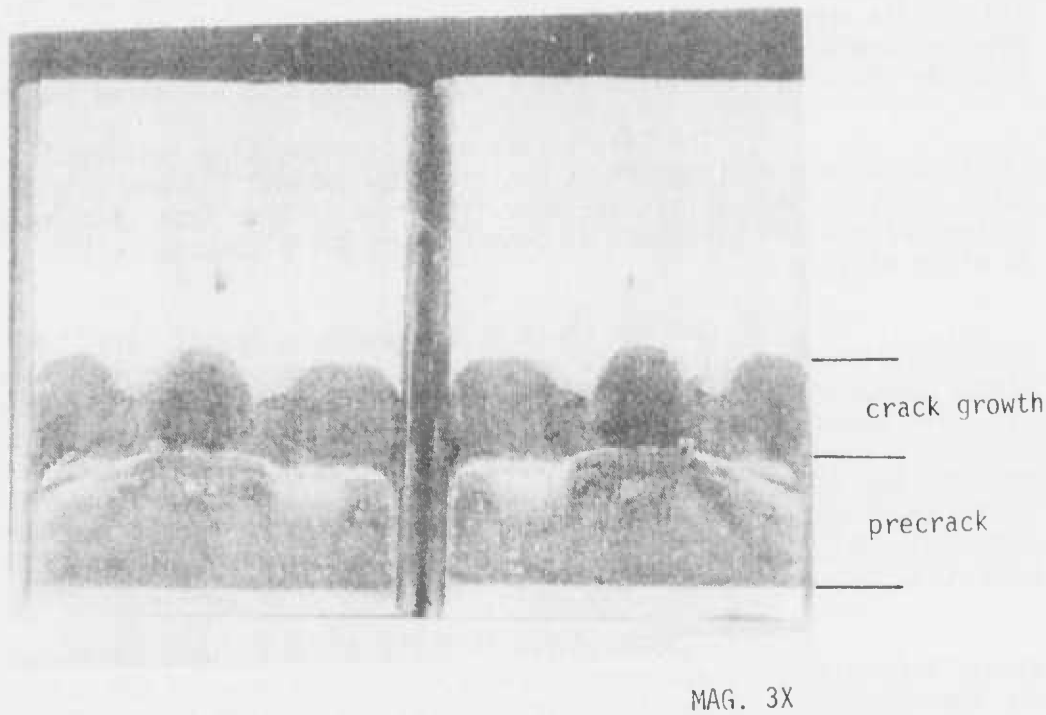


Figure 2.3.12. Welded 250 Maraging Steel Exposed to 2500 psia Gaseous NF_3 at 160°F

If the Titaniums, 6Al-4V and 5Al-2.5 Sn, are grouped together, a transition from no cracking in the welded Ti 5Al-2.5 Sn in liquid nitrogen trifluoride at -108°F to severe cracking in welded Ti 6Al-4V in 2500 psia gaseous nitrogen trifluoride at 160°F is seen. The increase in the stress corrosion cracking of the Titanium alloys as temperature and pressure of the nitrogen trifluoride is increased suggests that gaseous nitrogen trifluoride at 2500 psia is the most aggressive of the three test environments. The results with the welded CRES 17-4PH supports the aforementioned trend by showing stress corrosion cracking in the 2500 psia gaseous nitrogen trifluoride environment only, although it was tested in all three environments.

The best stress corrosion cracking resistance in the 160°F, 2500 psia environment is exhibited by Inconel 718 and C-1018 steel. These two iron base alloys are very different in chemistry with Inconel having major alloy additions of chromium and nickel whereas C-1018 steel has no alloy additions.

Another trend is indicated by the Al 2219, CRES 17-4PH, Ti 5Al-2.5 Sn and C-1018 steel specimens. In each of these materials the parent specimens stress corrosion cracked whereas the welded specimens did not. The general trend is for parent specimens to be more stress corrosion cracking susceptible than welded specimens. The difference in cracking behavior for welded versus parent specimens is attributed to the orientation of the crack plane in the rolled plate from which the parent specimens were machined. Welding would wipe out the effects of rolling direction in the weld metal but not in the heat affected zone.

K_i is included in Table 2.3-5 to show the reduction in stress intensity that accompanies crack growth. Because of the accuracy of the loading system it was impossible to set each stress corrosion cracking specimen at exactly K_i. However, a reduction in stress intensity from K_i to K_{ISCC} indicates crack growth that can be confirmed by examining specimen fracture surfaces as shown in Figure 2.3.12 and noted in the table entry for 250 Maraging Steel.

In general there is a good correlation between the K_{Iq} values from the fracture toughness tests and the stress corrosion cracking K_q values listed in Table 2.3-6. Exceptions are welded CRES 17-4PH in 160°F, 500 psia gas and -108°F liquid environments, parent Inconel 718 in the 160°F, 500 psia gaseous nitrogen trifluoride environment and the welded 250 Maraging steel in the 160°F, 2500 psia gaseous environment. In each of the aforementioned exceptions the K_q value for the specimens previously exposed to nitrogen trifluoride were higher than the K_{Iq} value which was exposed to air only.

2.3, Fracture Mechanics/Toughness Tests (cont.)

The Arde' 301 specimens, which were tested for qualitative information only, exhibited some crack growth in the 500 psia nitrogen trifluoride at 160°F. No K_{ISCC} value could be calculated.

The K_{ISCC} values obtained after the 180 days of exposure in some cases are not the final K_{ISCC} values; the crack growth was still occurring in some of the specimens. This is shown in Figure 2.3.13 for parent specimens of Al 2219 T-87 and CRES 17-4 PH in the 500 psia nitrogen trifluoride environment. The crack growth measurements are presented in Table 2.3-7 for the metal specimens which exhibited stress corrosion cracking during the exposure to nitrogen trifluoride.

In summation, Inconel 718 and 347 stainless steel were the only metals in which no stress corrosion cracking was detected. In addition, the stress corrosion cracking in C-1018 steel was found only to a slight extent in the parent specimens and not at all in the welded specimens. The titanium 6Al-4V specimens were found to be extremely susceptible to stress corrosion cracking.

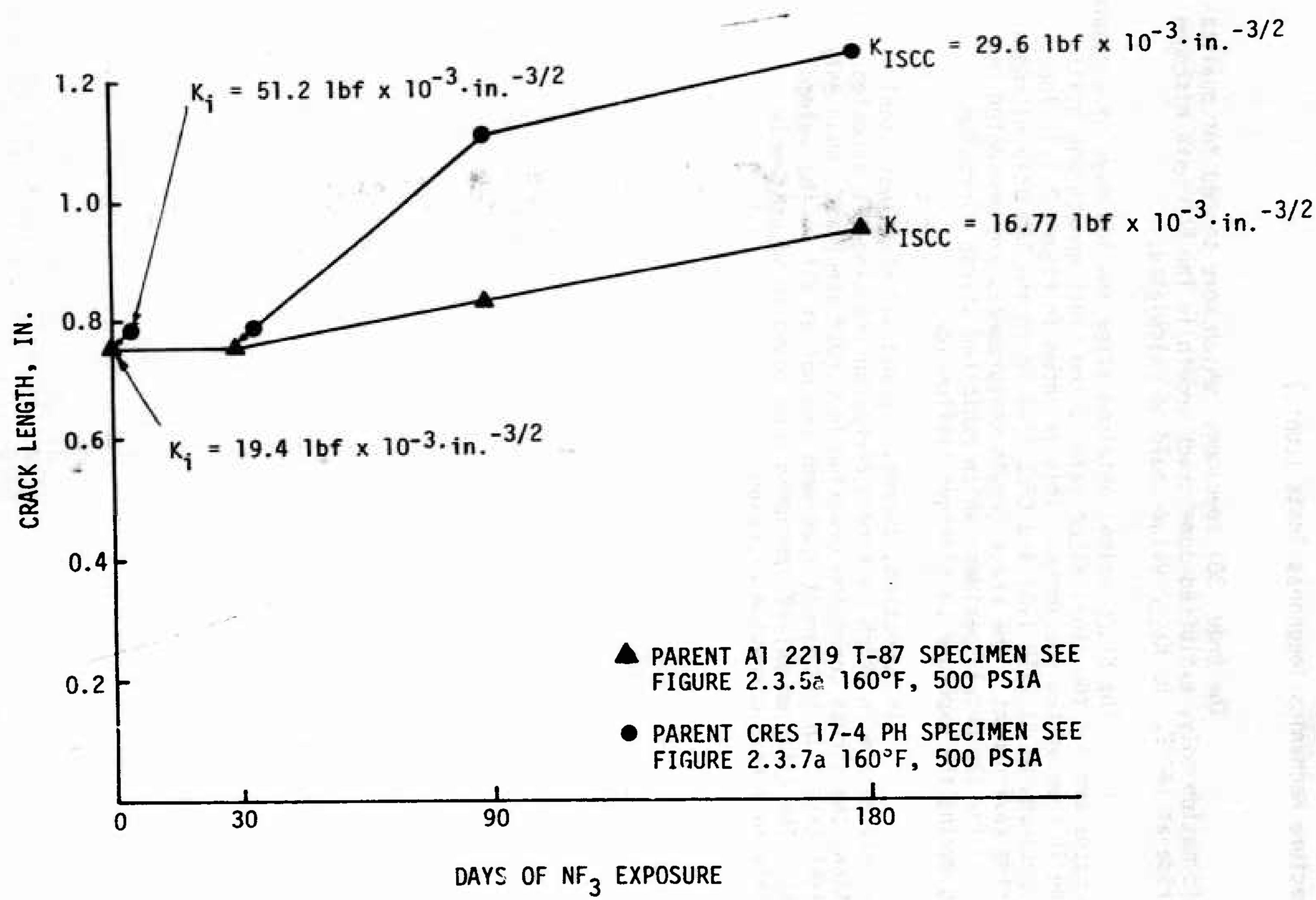


Figure 2.3.13. Crack Growth Length Versus Time of Exposure

TABLE 2.3-7

DATA INDICATIVE OF THE EXTENT OF CRACK GROWTH WHICH OCCURRED IN METAL SPECIMENS WHICH EXHIBITED STRESS CORROSION CRACKING

Material		NF_3 Environment	Specimen No.	Visually Measured Crack Length, in. 30 da	90 da	180 da	Initial Crack Length, in.	Crack Arrest Markes On Fracture Surface, Length in Inches 1	2	3
Al 2219-T87	Parent	160°F, 500 psia	1-1	0.772	0.783	0.874	0.776	0.766	0.942	
			1-2	0.702	0.750	0.793	0.728	0.728	0.870	
			1-3	0.766	0.764	0.793	0.761	0.761	0.876	
CRES 17-4 PH	Parent	160°F, 500 psia	3-1	0.728	1.053	1.128	0.756	0.756	0.988	1.237
			3-2	0.766	1.110	1.138	0.755	0.755	1.006	1.247
			3-3	1.041	1.173	1.219	0.814	0.814	1.256	1.300
CRES 17-4 PH	Welded	160°F, 2500 psia	3-6w	0.738	0.925	1.128	0.745	0.745	1.000	1.212
			3-10w	0.707	0.715	0.713	0.752	0.752	0.772	0.828
			3-11w	0.713	0.896	1.156	0.751	0.751	0.981	1.256
Ti 5Al-2.5 Sn	Parent	-108°F liquid	5-1	1.281	1.281	1.293	1.013	1.013	1.608	
			5-2	1.333	1.307	1.378	1.114	1.114	1.680	
			5-3	1.211	1.181	1.189	1.010	1.010	1.501	
Ti 6Al-4V	Parent	160°F, 500 psia	6-1	1.641	1.622	1.643	1.047	1.047	1.730	
			6-2	1.683	1.671	1.691	1.067	1.067	1.747	
			6-3	1.663	1.640	1.667	1.029	1.029	1.713	
			6-5	1.713	1.701	1.713	1.020	1.020	1.788	
			6-6	1.638	1.667	1.665	1.045	1.045	1.754	
Ti 6Al-4V	Parent	160°F, 2500 psia	6-4	1.703	Removed from Test After 30 da			--	--	
Ti 6Al-4V	Welded	160°F, 2500 psia	6-7w 6-8w	Cracked All the Way Through After 30 days				--	--	
C-1018 Steel	Parent	160°F, 2500 psia	7-1	1.069	0.998	1.026	0.968	0.968	1.214	
			7-2	0.953	0.965	0.965	0.936	0.936		
			7-3	0.972	0.990	0.984	0.956	0.956		
250 Marage	Welded	160°F, 2500 psia	8-1	0.732	0.756	0.758	0.764	0.764	0.831	
			8-2	0.771	0.939	0.939	0.841	0.841	1.030	
			8-3	0.752	0.772	0.783	0.787	0.787	0.805	

2.4 FLOW TESTS

The objective of the tests was to determine the maximum temperature levels that materials can withstand in flowing gaseous nitrogen trifluoride for short periods of time without the materials exhibiting detrimental effects and to determine whether the detrimental effects were dependent on the velocity of the gas.

Eleven representative metals and four non-metallic materials were tested to define the threshold non-ignition temperatures for nitrogen trifluoride/material interactions and the effect of velocity on the threshold values. The materials tested were as follows:

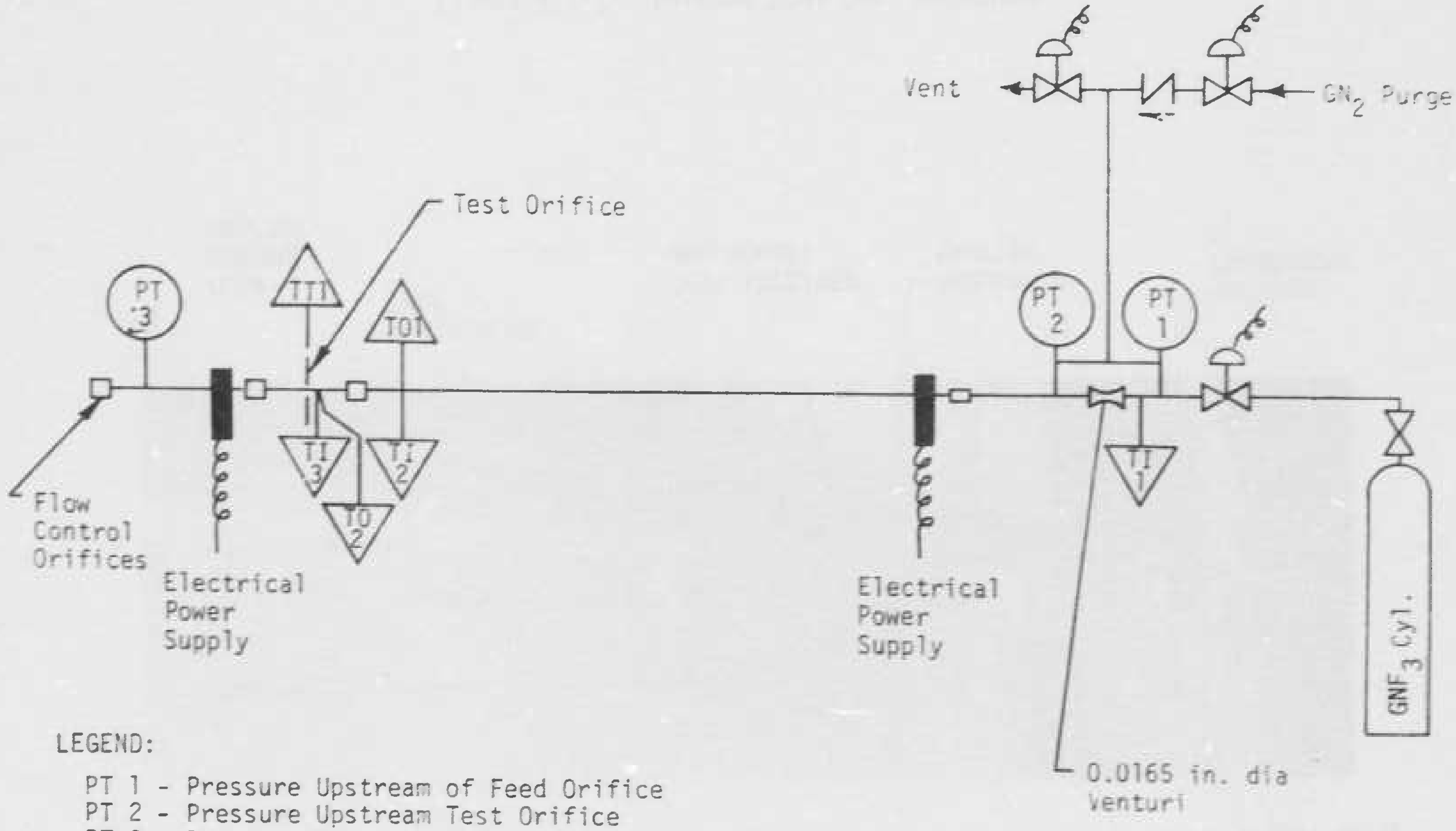
Al 2219, T-87	Nickel 200, Annealed	CRES 17-4PH, H-1025
CRES 304L, Annealed	Ti 6 Al-4V, STA	PFA Teflon
CRES 316L, Annealed	1018 Steel	Polytetrafluoroethylene
Inconel 625, Annealed	Cu OFHC Annealed	Kel-F 81 CTFE
Monel 400, Annealed	Narloy A	Carbon CJPS

One series of tests was conducted in the subsonic velocity regime, and one series was conducted in the sonic velocity regime.

2.4.1 Apparatus and Procedures

A schematic diagram of the apparatus in which the tests were conducted is shown in Figure 2.4.1; a photograph of the entire apparatus is shown in Figure 2.4.2. The basic apparatus consists of an Inconel X-750 tube, 1.27 cm (0.5 inch) diameter with .89 mm (.035 inch) wall thickness which was heated by direct electrical resistance heating. The tube is divided into three parts: a preheat section, the heated test section containing the metal specimen of choice, and a downstream section with the flow controlling orifice.

The test section is approximately 15.2 cm (6 in.) long. A cylinder of the metal to be tested is welded in place between two sections of the Inconel X-750 tubing. A .508 mm (0.020 inch) diameter hole was drilled through the metal specimen and the length of the hole was approximately .64 cm (0.25 inches). An isolated junction, chromel-alumel thermocouple sheathed in .508 mm (0.020 inch) diameter Inconel was inserted into a hole drilled into the side of the metal specimen to within .25 mm (0.010 inch) of the flow passage. An assembled test section is shown in Figure 2.4.3 with the thermocouples in place. The metal specimens which could not be readily welded to the Inconel, i.e., Copper, Narloy A, 2219 Al, and Titanium 6Al-4V and the non-metal specimens were swaged in place in the Inconel tube and fitted with thermocouples similarly.



LEGEND:

PT 1 - Pressure Upstream of Feed Orifice
PT 2 - Pressure Upstream Test Orifice
PT 3 - Pressure Downstream Test Orifice

TI 1 - Temperature of Gas Supply
TI 2 - Temperature of the Heated Gas
TO 1 - Temperature of Tube Surface
TI 3 - Temperature of Gas at Test Orifice Face
TO 2 - Temperature of Tube Surface at Test Orifice
TT 1 - Temperature of the Test Orifice

Figure 2.4.1. Schematic of Gaseous Flow Test Apparatus

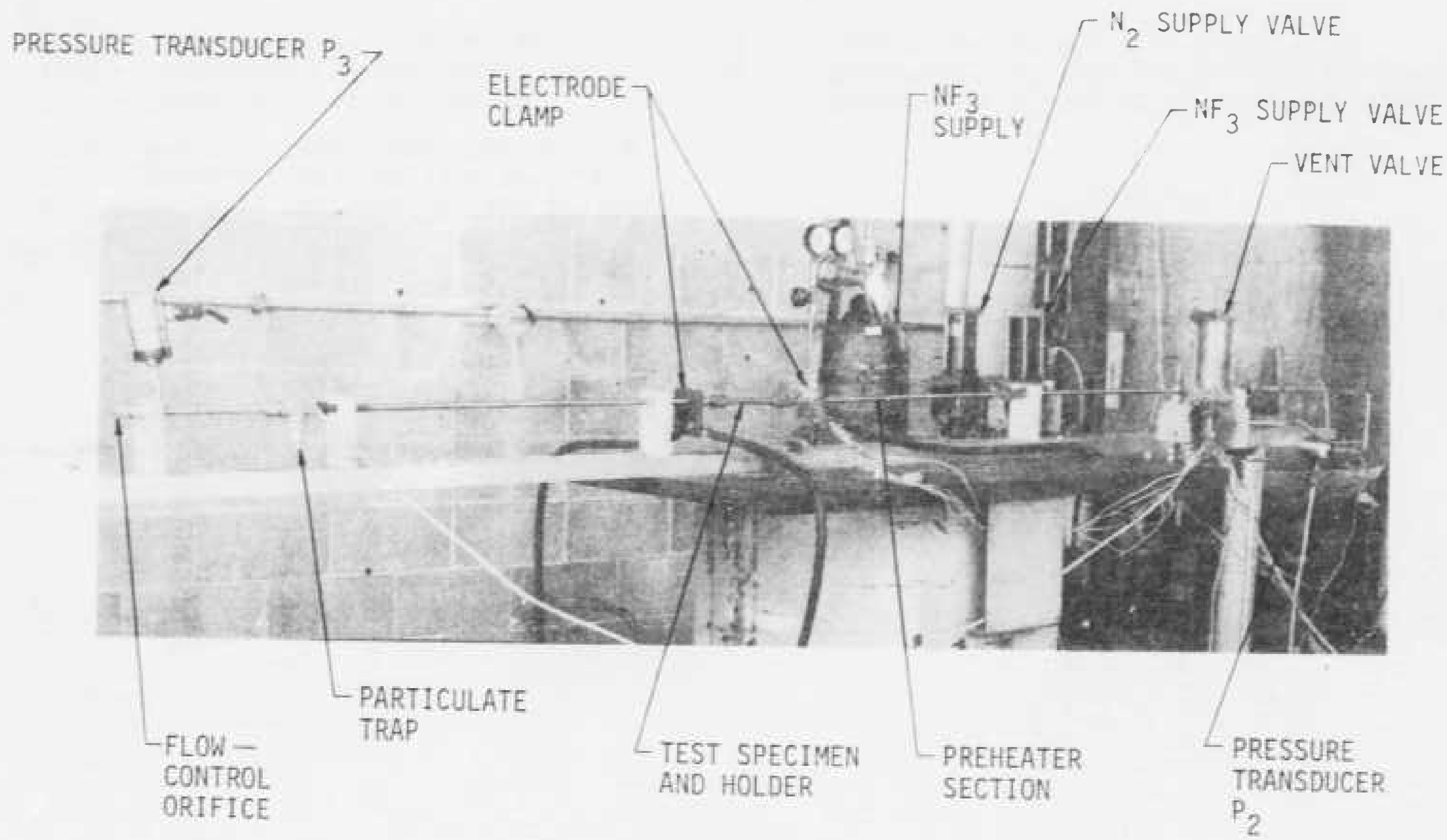


Figure 2.4.2. Gaseous Flow Test Apparatus

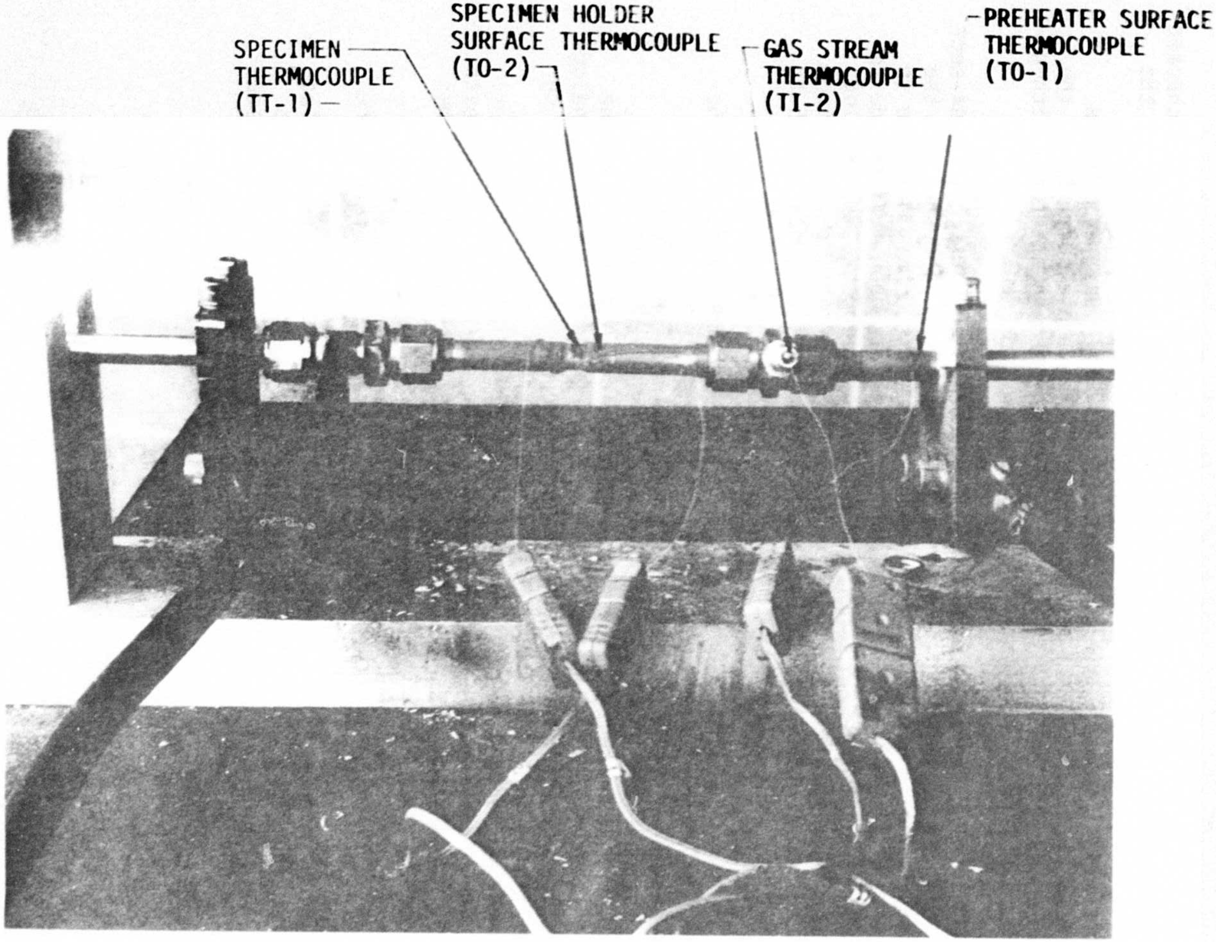


Figure 2.4.3. Flow Test Specimen and Holder With Thermocouples Attached

Prior to conducting the tests, the apparatus was checked out using nitrogen to insure satisfactory operation and calibration of the test apparatus. A special specimen holder was fabricated which had a thermocouple TI-3 located in the gas stream immediately upstream of the orifice. A comparison of the readings of the thermocouple inserted in the sample (TT-1) and the one located in the gas stream showed agreement within 8 K (15 F).

The procedure for testing was as follows. The test section containing the desired metal disc was placed in the apparatus and the system was purged with nitrogen to insure that the test conditions were in order. Then the system was flushed repeatedly with nitrogen trifluoride to remove the nitrogen. Then with nitrogen trifluoride flowing, the heating of the apparatus was initiated. The pressures used in testing ranged from .15 to 1.5 MN/m^2 (22 to 220 psia) and the heating rates ranged from 5.5 to 11 K (10 to 20 F)/sec.

The flow rate of the gas through the orifice of the test specimen was controlled by the use of orifices at the exit of the downstream section for the subsonic tests and for the sonic velocity tests the exit was left open. In case of burn out of the test section, the gaseous flow was limited by a .42 mm (0.0165-in.) dia venturi in the unheated portion of the feed system. The data were recorded with an analog-to-digital recorder with each instrumentation channel sampled 85 times per second (the system sampling rate is 3,750 channels per second) and the data were reduced using a Hewlett Packard Model 2100 electronic computer. A direct read-out system and television monitor were used during the tests as means to indicate heating rates and to indicate when the test specimen failed.

The pressure transducers used in the apparatus were accurate to $\pm$ 4.1 kN/m^2 ($\pm$ 0.6 psi) and were sensitive to 0.69 kN/m^2 (0.1 psi) which is more than adequate for the testing. The thermocouples were prepared from special chromel and alumel wire and were accurate to $\pm$ 0.38% in the temperature range that was encountered. Thus, the temperature values reported are accurate to at least ($\pm$ 10°F).

2.4.2 Experimental Results

The nitrogen trifluoride used in the gaseous flow tests was analyzed and the composition was as follows.

2.4, Flow Tests (cont.)

Component	Weight Percent
NF_3	99.68
Active fluoride as HF	.0003
CO/O_2	0.29
CF_4	0.017
N_2O	0.014

The analysis was conducted with and without the KI scrubber in the gas chromatograph sampling loop and the results were in agreement.

Examples of the swaged specimen holders used in the testing are shown in Figure 2.4.4; examples of the welded specimen holders used in the testing are shown in Figure 2.4.5.

Typical plots of the data which were obtained from the tests are presented in three figures. In Figure 2.4.6 the initial velocity through the titanium 6Al-4V orifice was subsonic, the pressure ratio being 1.23. At the temperature labeled "1", there is the onset of a major film buildup as evidenced by the increase in the pressure drop across the orifice and the increase in the pressure ratio across the orifice due to the decrease in the orifice diameter. At the temperature labeled "2" there is a slight disruption in the pressure increases indicative of the loss of a small amount of material. At the temperature labeled "3", a major exotherm occurs as evidenced by sudden temperature increases and there is significant loss of materials as evidenced by the sudden decrease in pressure drop across the test orifice.

In Figure 2.4.7, the initial velocity through the nickel 200 test orifice is subsonic, the pressure ratio being 1.09. No significant events occurred until the temperature reached the position labeled "1", at which time a film formation occurred in the orifice as evidenced by a pressure drop increase and an endothermic reaction. At the temperature labeled "2", there was a minor loss of film as evidenced by the reversal of the pressure drop trend. At the temperature labled "3", some gradual film loss occurred as shown by a gradual decrease in the pressure drop; at the temperature labled "4" the film formation process was again repeated.

In Figure 2.4.8, the initial velocity through the copper OFHC was subsonic, the pressure ratio being 1.17. At the temperature labeled "1", there is the gradual onset of some film formation as evidenced by an increase in pressure drop across the orifice. At the temperature

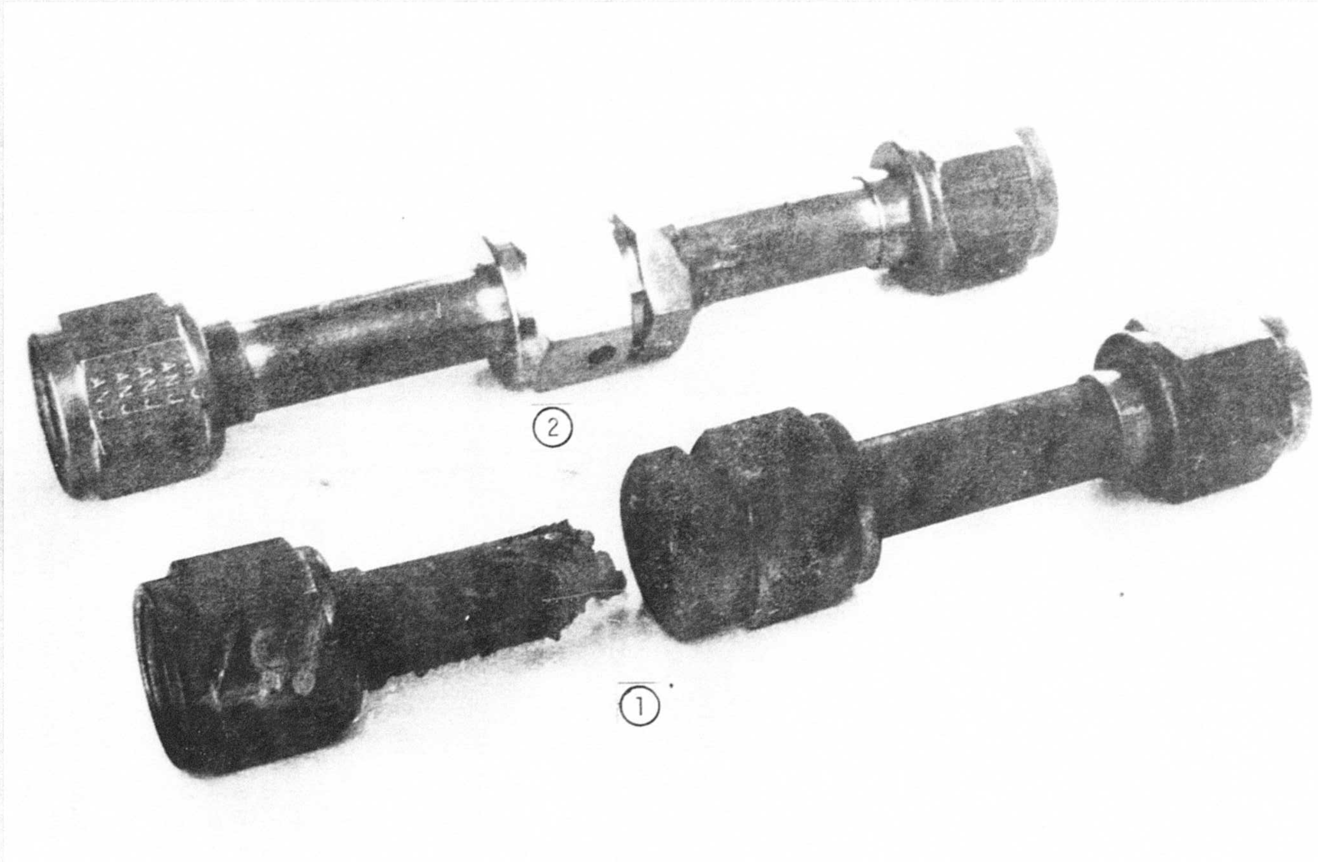


Figure 2.4.4. Swaged Specimens Holders: (1) Heated to Failure, (2) Heated Until Threshold Events Occurred

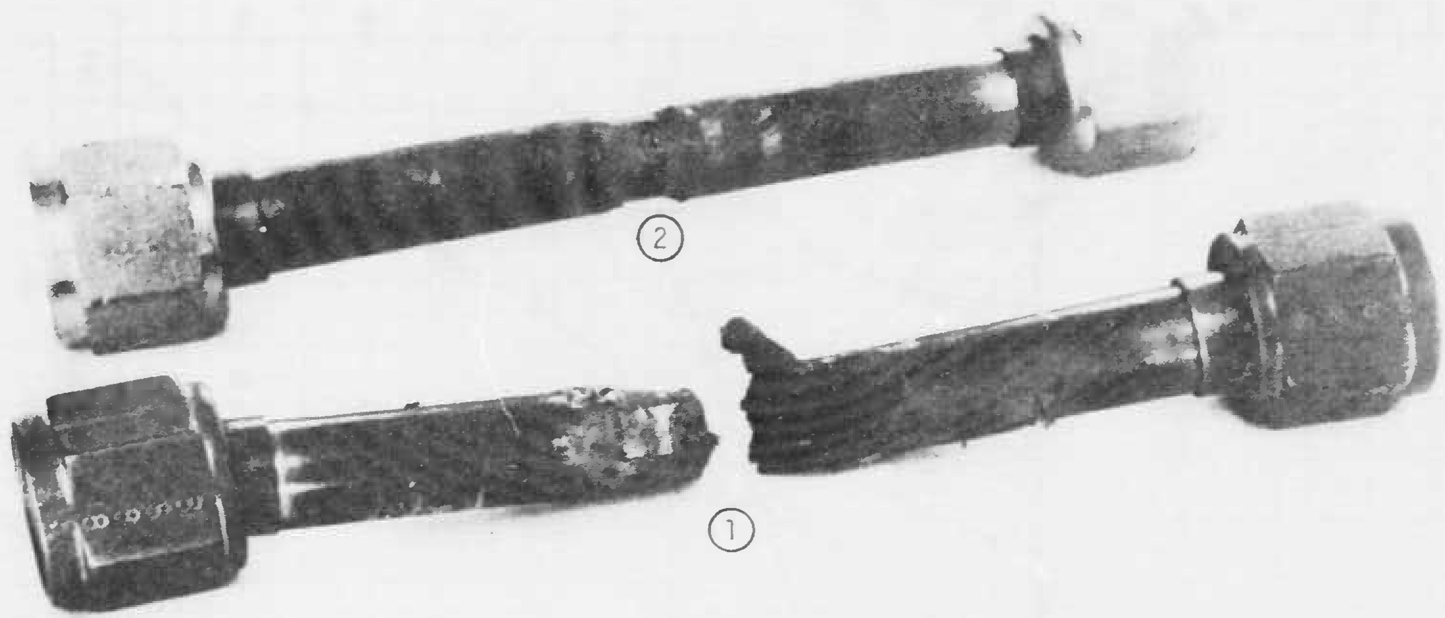


Figure 2.4. 5. Welded Specimen Holders: (1) Heated to Failure (2) Heated Until Threshold Events Occurred

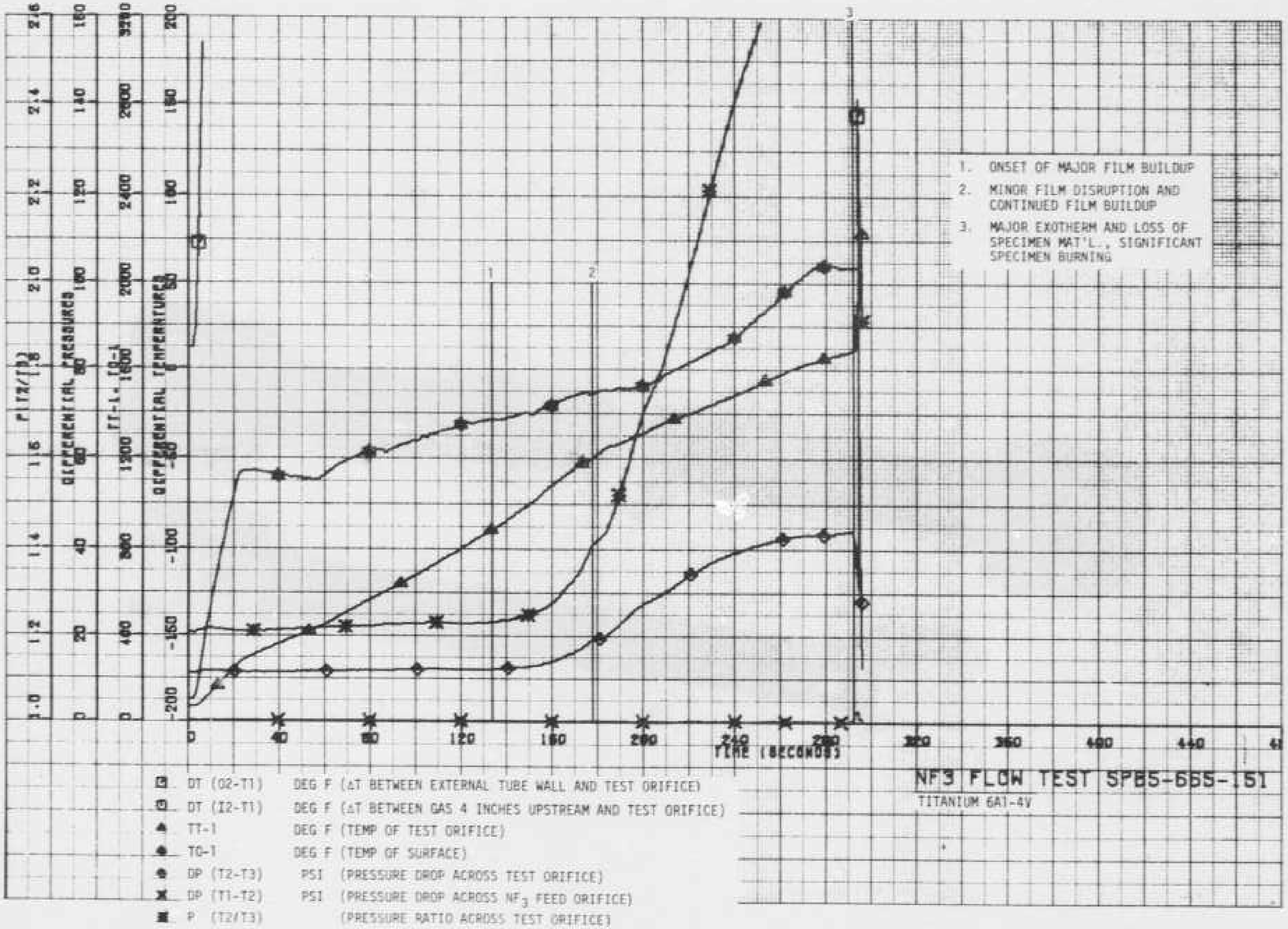


Figure 2.4.6. Plot of Data Obtained from Gaseous Flow Test with 6 Al-4V Titanium Specimen

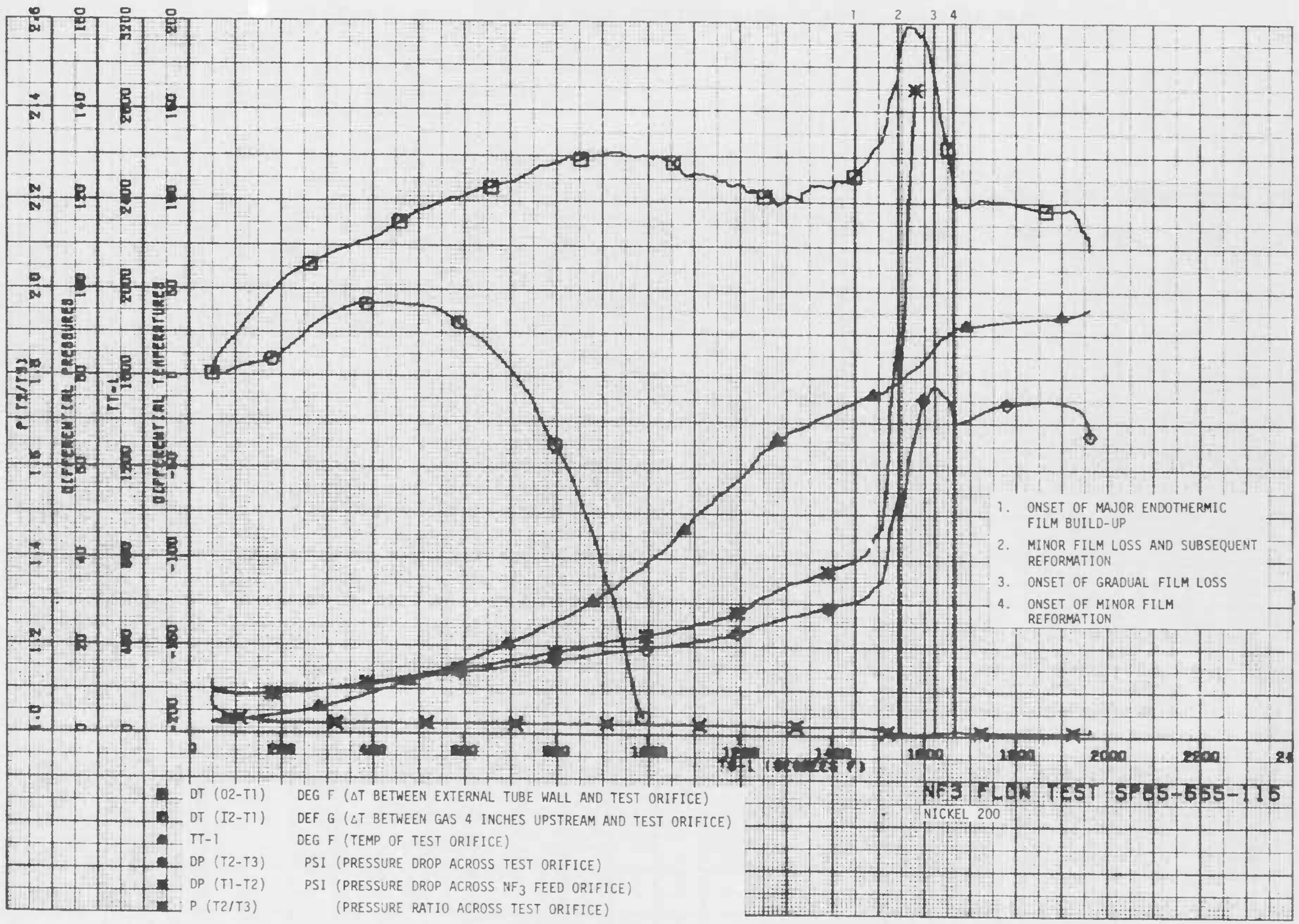


Figure 2.4.7. Plot of Data Obtained from Gaseous Flow Test with Nickel 200 Specimen

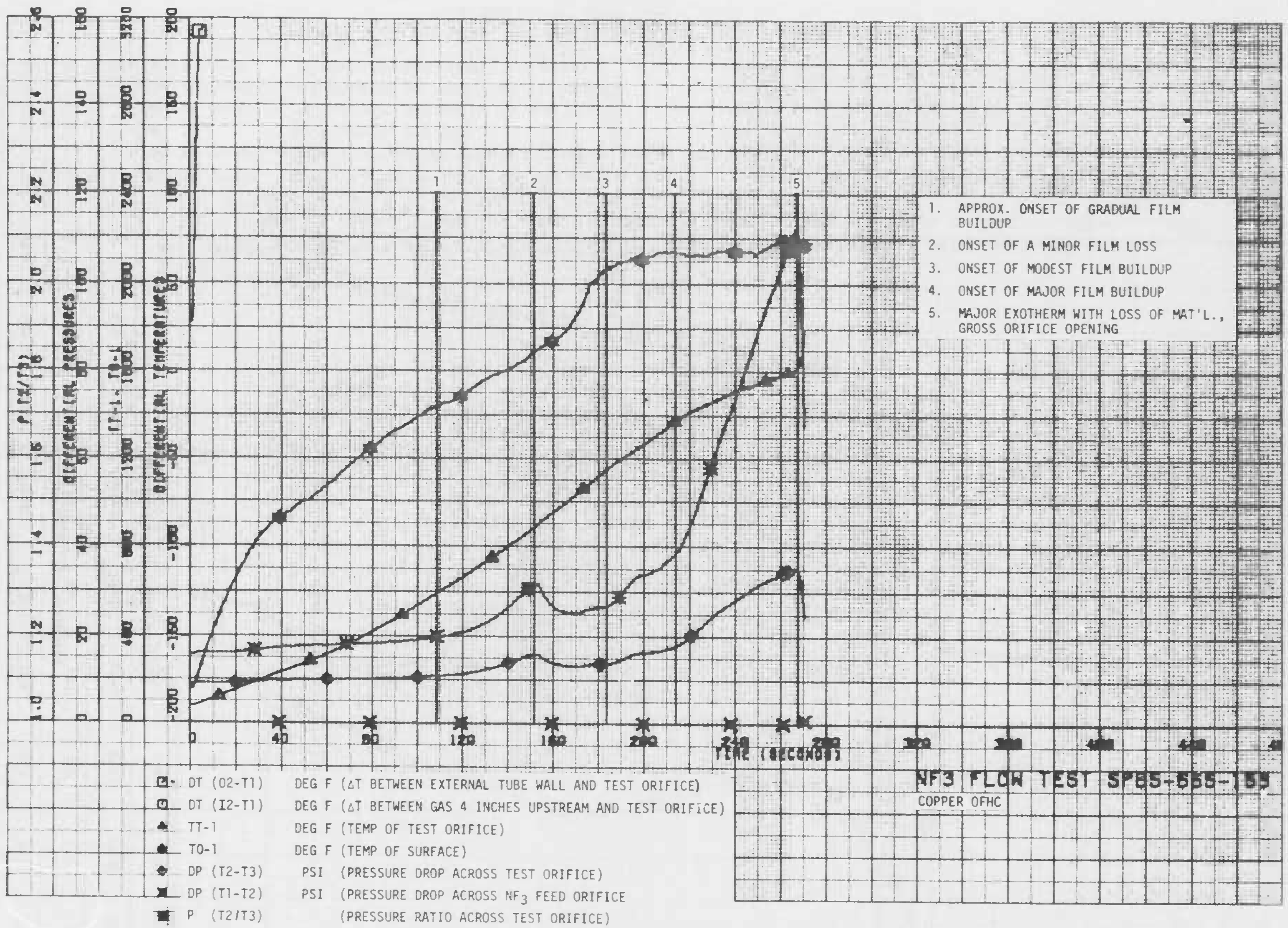


Figure 2.4.8. Plot of Data Obtained from Gaseous Flow Test with Copper OFHC Specimen

labeled "2", some minor film loss is noted by the decrease in pressure drop. At the temperature labeled "3", the film formation reoccurs as evidenced by an increase in the pressure drop. At the temperature labeled "4", the pressure drop increases more rapidly and the pressure ratio increases rapidly which is indicative of some major film formation in the test orifice. At the temperature labeled "5", a major exotherm occurs with a concurrent decrease in the pressure drop which is indicative of the loss of a significant amount of material.

The test data obtained and interpreted in the manner described above are presented in Table 2.4-1 for the metallic materials and in Table 2.4-2 for the non-metallic materials. In the case of the tests initiated at subsonic velocities the pressure ratio across the test orifice is indicated; in the case of tests at sonic velocity (pressure ratio $\geq$ 1.78) the pressure ratio is indicated as "sonic".

From the data in Tables 2.4-1 and 2.4-2 it is not apparent that the velocity significantly affects the temperature at which nitrogen trifluoride/material interactions occur. Generally higher pressures and sonic velocities did intensify the reactions which occurred. From the data, three threshold temperature values have been identified. The first attack threshold is the lowest temperature at which the material under any of the conditions of pressure and velocity investigated gave a detectable response attributable to attack. The second threshold temperature, referred to as "major-corrosion", is the lowest temperature under any of the conditions investigated at which major corrosion as evidenced by large thermal or pressure changes occurred. The third threshold temperature, referred to as the incipient-failure threshold, is the temperature at which gross material failure is first detected. The various threshold temperatures for each of the materials is presented in Table 2.4-3.

In considering this data for design limits one should keep in mind that the events detected occurred within the time frame of seconds and that long-term exposure at temperatures below the threshold values may result in intolerable corrosion rates or other unacceptable behavior.

It is of some interest to observe that the first-attack threshold for copper given in Table 2.4-3 (505 K) compares very favorably with the onset of an exothermic reaction at approximately 498 K observed by Pisacone, et al. (Reference 2.4-1) who employed a differential scanning calorimeter and low-pressure gaseous NF_3 under static conditions. They also observed further but stronger exotherms at 543 and 613 K which would appear to correspond roughly to the major-corrosion threshold for copper (589 K) given in Table 2.4-3. Pisacane's study of aluminum showed an

TABLE 2.4-1

DATA INDICATIVE OF THE BEHAVIOR OF VARIOUS METALS WITH FLOWING GASEOUS NITROGEN TRIFLUORIDE AT ELEVATED TEMPERATURES

Material	Specimen Temperature °K	Specimen Temperature °F	Orifice Pressure Ratio	Upstream Pressure MN/m^2	Upstream Pressure psia	Material Response	Test No.
304-L	1094	1510	Sonic	.67	97	Minor film buildup	104
Stainless	1094	1510		.78	113	Moderate film buildup	112
Steel	1133	1580		.79	115	Moderate film loss	112
	1144	1600		.69	100	Major film buildup	104
	1155	1620		.78	113	Major film buildup	112
	1197	1695		.77	112	Major exotherm with loss of mat'l.	104
	1197	1695		.83	120	Major exotherm with loss of mat'l., gross specimen burning	112
	878	1120	1.44	.40	58	Very minor film loss and reformation	157
	1158	1625	1.49	.40	58	Major film buildup	157
	1200	1700	Sonic	.42	61	Major loss of mat'l., orifice enlarged appreciably	157
316 ELC	1100	1520	Sonic	1.21	175	Major film buildup	107
Stainless	1158	1625		1.25	181	Film loss	107
Steel	1172	~1650		1.24	180	Film buildup	107
	1203	1705		1.25	182	Major exotherm and loss of mat'l., gross specimen burning/ melting	107
	914	1185	1.60	.57	83	Minor film loss	103
	955	1260	1.44	.58	84	Minor film buildup	103
	964	1275	Sonic	.79	115	Minor film buildup	111
	1066	1460		.59	86	Minor film loss	103
	1100	1520		.61	88	Major film buildup	103
	1103	1525		.80	116	Major film buildup	111
	1139	1590		.81	118	Minor film loss	111
	1158	1625		.79	114	Minor film loss	103
	1180	1665		.73	106	Minor film buildup	103
	1205	1710		.76	111	Major exotherm and loss of mat'l., gross specimen burning	103
	1208	1715		.83	120	Major exotherm and loss of mat'l., gross specimen burning	111
	1144	~1600	1.49	.40	58	Major film buildup	156
	1241	1775	Sonic	.43	62	Major exotherm and loss of mat'l., moderate specimen burning	156
17-4PH,	1155	1620	Sonic	1.18	171	Significant exotherm film buildup	106
H-1025	1178	1660		1.20	174	Major exotherm with loss of specimen mat'l., gross specimen burning	106
	1139	1590	1.46	7.65	111	Minor exotherm and film buildup	114
	1166	1640	1.50	7.65	111	Major exotherm with loss of spec. mat'l., excess specimen burning	114
	866	~1100	1.31	.41	60	Very minor material loss	158
	1005	~1350	1.32	.41	60	Onset of a minor film buildup	158
	1122	~1560	1.36	.41	60	Major film buildup	158
	1178	1660	Sonic	.43	63	Major exotherm with loss of spec. mat'l.	158
1018 Carbon	750	~890	Sonic	1.37	198	Small, sharp endotherm	138
Steel	1089	~1500		1.44	209	Onset of major film loss	138
	1133	1580		1.43	208	Major exotherm with loss of spec. mat'l., gross orifice burning	138
	894	~1150	1.35	.73	106	Onset of appreciable buildup	117
	1000	~1340	1.56	.75	109	Modest endotherm	117
	1080	1485	Sonic	.76	110	Rapid endothermic film buildup	117
	1100	1520		.77	111	Minor film loss and subsequent major reformation	117
	1144	1600		.79	115	Major exotherm with loss of spec. mat'l., gross orifice burning	117
	908	~1175	1.39	.14	20	Onset of minor film buildup	145
	1061	~1450	1.40	.14	20	Modest exothermic film loss	145
	1311	1900	1.34	.13	19	Moderate film buildup	145
	1386	2035	1.37	.14	20	No failure to max. test temp., some orifice opening observed	145
Monel 400	889	~1140	1.30	.76	110	Minor sharp exotherm	113
	903	~1165	Sonic	.61	89	Minor sharp exotherm	105
	1058	~1445	Sonic	.64	93	Moderate exothermic film buildup	105
	1066	~1460	1.41	.76	110	Moderate exothermic film buildup	113
	1116	1550	1.45	.76	110	Major film buildup	113
	1141	1595	Sonic	.81	117	No failure to max. test temp., orifice plugged off	113
	1144	1600		.66	96	Major film buildup	105
	1178	1660		.70	101	Very rapid endothermic film buildup	105
	1214	1725		.76	110	Minor film loss and reformation	105
	1300	1880		.81	117	Minor film loss	105
	1311	1900		.80	116	No failure to max. test temp., orifice essentially plugged off	105
	1075	1475	1.63	.38	55	Onset of moderate film buildup	155
	1125	1565	Sonic	.39	57	Major endothermic film buildup	155
	1144	1600		.41	59	Minor film loss followed by continuing film buildup	155
	1286	1855		.45	65	No failure to max. test temp. but orifice essentially plugged off	155

TABLE 2.4-1 (cont.)

Material	Specimen Temperature °K	Specimen Temperature °F	Orifice Pressure Ratio	Upstream Pressure MN/m^2	Upstream Pressure psia	Material Response	Test No.
Inconel 625	950	1250	Sonic	1.38	200	Apparent onset of a slow, minor exotherm	139
	1116	1550		1.39	202	Major endotherm with some film buildup	139
	1194	1690		1.42	206	Modest film loss followed by film reformation	139
	1244	1780		1.42	206	Modest film loss followed by film reformation	139
	1264	1815		1.41	204	Modest film loss followed by film reformation	139
	1294	1870		1.41	205	Minor film loss followed by film reformation	139
	1305	1890		1.43	207	Modest film loss followed by film reformation	139
	1372	2010		1.53	222	No failure but essentially complete blockage of orifice	139
	1466	2180	---	1.53	222	Burnout failure of preheater section	139
	1044	1420	1.34	.72	105	Modest film buildup	118
	1075	1475	1.46	.73	106	Modest film loss	118
	1114	1545	1.39	.72	105	Major endothermic film buildup	118
	1172	1650	Sonic	.80	116	Minor film loss followed by film reformation	118
	1222	1740		.77	112	Minor film loss followed by film reformation	118
	1239	1770		.77	111	Minor film loss	118
	1255	1800		.75	109	No failure to maximum test temperature, orifice virtually unchanged	118
Nickel 200	1061	~1450	Sonic	1.35	196	Significant film buildup	140
	1150	~1610		1.39	201	Moderate film loss and endothermic reformation	140
	1166	1640		1.38	200	Minor film loss and endothermic reformation	140
	1200	1700		1.39	201	Minor film loss and subsequent buildup	140
	1266	1820		1.44	209	Minor film loss and subsequent buildup	140
	1297	1875		1.44	209	Minor film loss and subsequent buildup	140
	1308	1895		1.44	209	Minor film loss and subsequent buildup	140
	1350	1970		1.50	218	No failure to max. test temp., orifice essentially plugged off	140
	1083	1490	1.40	.74	107	Major endothermic film buildup	116
	1133	1580	Sonic	.78	113	Minor film loss and subsequent reformation	116
	1172	1650		.79	115	Onset of a gradual film loss	116
	1269	1825		.75	109	Onset of a minor film reformation	116
	1322	1920		.74	108	No failure to max. test temp., orifice open and virtually unchanged	116
Copper OFHC	505	450	Sonic	.62	90	Very minor film loss and reformation	142
	561	550		.62	90	Minor film loss	142
	589	600		.63	91	Significant film buildup	142
	622	660		.64	93	Minor film loss and reformation	142
	866	1100		.63	92	Slow film buildup	142
	916	1190		.67	97	No failure to maximum test temp.	142
	589	600	1.20	.46	66	Minor film buildup	153
	750	890	1.32	.46	67	Minor film loss and reformation	153
	905	1170	1.27	.45	65	Moderate film buildup	153
	1022	1380	1.39	.44	64	Major film buildup	153
	1161	1630	Sonic	.46	66	Major exotherm with loss of mat'l., gross orifice opening	153
	589	600		.19	28	Very minor film loss	143
	714	825		.18	26	Possible minor film formation	143
	811	1000	1.62	.16	23	Possible minor film loss	144
	911	1180	1.58	.16	23	Minor film buildup	144
	983	1310	Sonic	.20	29	No failure to maximum test temp.	143
	1108	1535	1.70	.17	24	Moderate film loss	144
	1158	1625	1.59	.16	23	Moderate film loss	144
	1203	1705	1.57	.16	23	No failure to maximum test temp., orifice enlarged appreciably	144
Narloy A	958	1265	Sonic	1.21	176	Minor film loss	110
	1011	1360		1.21	175	Moderate exothermic film loss	110
	1028	1390		1.18	171	Major exotherm with loss of mat'l., gross specimen burning	110
	822	1020	1.27	.48	70	Very minor film loss	147
	897	1155	1.26	.48	69	Onset of moderate film buildup	147
	905	1170	1.23	.45	65	Onset of rapid film buildup	152
	939	1230	1.30	.48	69	Major film buildup	147
	953	1255	1.37	.46	67	Modest film loss	152
	983	1310	1.28	.45	65	Major film buildup	152
	1019	1375	Sonic	.50	72	Orifice totally plugged off	147
	1189	1680		.46	66	Maximum film buildup achieved and onset of a significant film loss	152
	1230	1755		.44	64	Major exotherm with loss of mat'l., gross orifice opening	152
6Al-4V Titanium	744	~880	1.25	.49	71	Onset of film buildup	146
	750	~890	1.23	.44	64	Onset of film buildup	151
	922	1200	1.41	.46	66	Minor film disruption and subsequent rebuilding	151
	941	1235	1.33	.49	71	Modest film loss followed by more rapid film buildup	146
	983	~1310	1.70	.46	67	Small decrease in film buildup rate	151
	989	1320	1.45	.50	73	Minor film loss and subsequent rebuilding	146
	1091	1505	Sonic	.51	74	No failure to max. test temp., orifice plugged off	146
	1191	1685		.46	66	Major exotherm and loss of spec. mat'l., significant specimen burning	151
2219 Aluminum, T-87	658	~725	1.27	.43	63	Onset of gradually increasing rate of film buildup	148
	908	~1175	Sonic	.45	65	Orifice essentially plugged off, specimen partially melted along holder boundary	148

TABLE 2.4-2

DATA INDICATIVE OF THE BEHAVIOR OF SELECTED NON-METALLIC MATERIALS WITH FLOWING GASEOUS NITROGEN TRIFLUORIDE AT ELEVATED TEMPERATURES

Material	Specimen Temperature °K	Specimen Temperature °F	Orifice Pressure Ratio	Upstream Pressure N/m^2	Upstream Pressure psia	Material Response	Test No.
Kel-F 81	458	~365	Sonic	1.30	188	Sudden small increase in flow area accompanied by an exotherm	108
	489	420		1.24	180	Rapid gross increase in flow area	108
	508	455		0.99	143	Onset of an exothermic flow restriction	108
	528	490		1.01	146	Specimen broke loose from its holder without specimen failure	108
	525	485		0.88	127	Onset of a increase in flow area	115
	547	525		0.85	124	Specimen broke loose from its holder, evidence of melting but no charring	115
Polytetra-fluoroethylene	489	~420	Sonic	1.33	193	Abrupt increase in flow area	109
	536	505		.96	139	Onset of a mildly endothermic flow restriction	109
	766	920		1.05	153	Orifice melted closed, spec. melted at holder boundary and slid downstream, some specimen discoloration	109
	489	~420	1.52	.44	64	Onset of a gradually increasing flow restriction	149
	802	985	Sonic	.48	70	Major exotherm and loss of specimen	149
PFA Teflon	580	585	Sonic	.47	68	Onset of a strong exotherm, spec. melted at orifice and holder boundary	150
Carbon CJPS	844	1060	1.56	.81	118	Brief exotherm without significant orifice dimensional change	141
	1011	1360	1.64	.81	117	Onset of major exothermic loss of spec. mat'l.	141
	1172	~1650	1.35	.77	111	Onset of apparent orifice constriction accompanying an exothermic reaction	141
	1289	~1860	1.43	.77	111	Endothermic reaction and apparent continuing orifice constriction	141
	1366	2000	1.59	.77	112	Major exotherm, gross specimen burning	141
	1078	1480	1.15	.46	66	Onset of apparent major orifice constriction accompanied by exothermic reaction	154
	1186	~1675	1.52	.46	67	Apparent endothermic reaction and continuing orifice restriction	154
	1272	~1830	1.58	.46	66	Apparent exothermic reaction and continuing orifice restriction	154
	1339	1950	1.66	.46	67	No gross failure to max. test temp., orifice plugged off	154

TABLE 2.4-3

THRESHOLD REACTION TEMPERATURES OF MATERIALS SUBJECTED TO SHORT-TERM, HIGH-VELOCITY FLOW OF COMPRESSED GASEOUS NF_3

	Threshold Temperatures					
	First Attack		Major Corrosion		Incipient Failure	
Material	K	F	K	F	K	F
Nickel 200	~1061	~1450	-	-	>1350	>1970
Inconel 625	950	1250	1114	1545	>1372	>2010
316 L Stainless Steel	914	1185	1100	1520	1203	1705
Monel 400	~889	~1140	1116	1550	>1311	>1900
304 L Stainless Steel	878	1120	1144	1600	1197	1695
17-4 PH Stainless Steel	~866	~1100	~1122	~1560	1166	1640
Narloy A	822	1020	905	1170	1028	1390
1018 Carbon Steel	~750	~890	1080	1485	1133	1580
Titanium 6 Al-4V	~744	~880	922	1200	1191	1685
Aluminum 2219	~658	~725	-	-	~908[(a)]	~1175[(a)]
Copper OFHC	505	450	589	600	1161	1630
Carbon CJPS	844	1060	1011	1360	1366	2000
Polytetrafluoroethylene	~489	~420	-	-	766[(a)]	920[(a)]
PFA Teflon	-	-	-	-	580[(a)]	585[(a)]
Kel-F 81 CTFE	~458	~365	489	420	528[(a)]	490[(a)]

(a) Onset of softening/partial melting.

2.4, Flow Tests (cont.)

exotherm at about 600 K. That reaction probably is related to the first-attack threshold for aluminum ($\sim$ 658 K) as given in Table 2.4-3. It can thus be seen that the thresholds defined under flow conditions mark significant NF_3/material interactions that, at least in some cases, correspond reasonably well with interactions observed under static conditions.

2.0, Experiment Results and Discussion (cont.)

2.5 ADIABATIC COMPRESSION TESTS

The objective of the adiabatic compression tests is to determine the behavior of selected materials in the presence of nitrogen trifluoride subjected to adiabatic compression. The test simulates the condition which can occur during the rapid opening and closing of valves. The materials selected for testing in the tabulation below are considered to be representative system materials which could be exposed to NF_3 undergoing rapid compression.

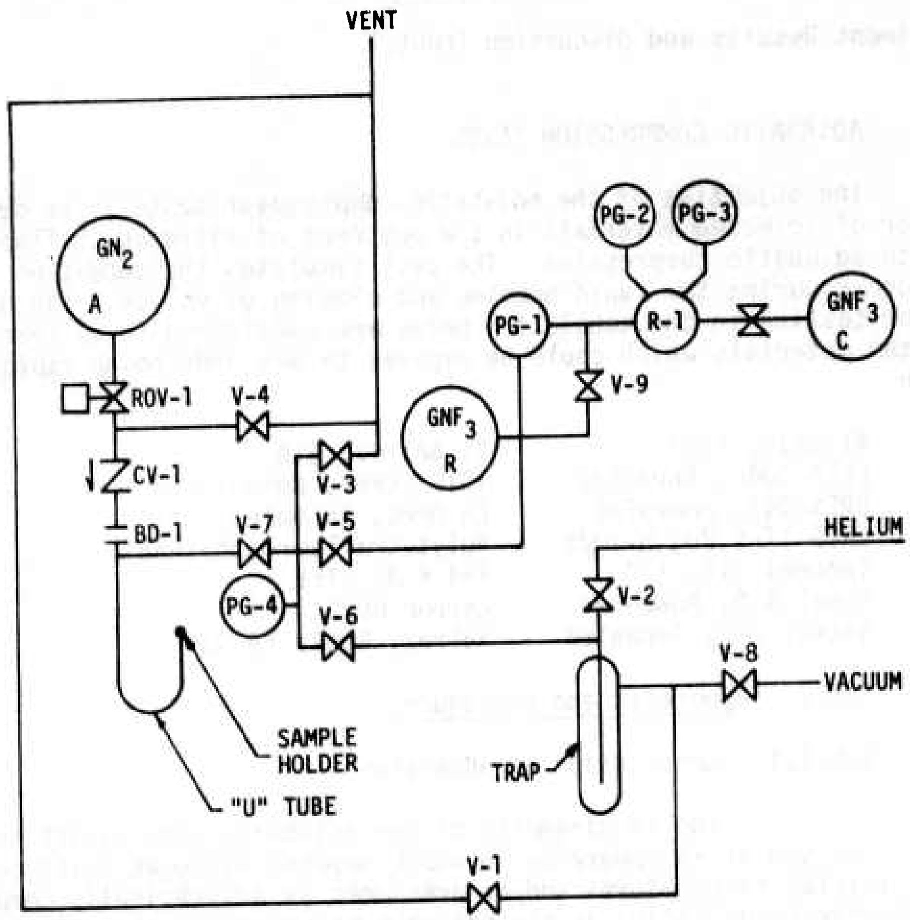
Al 2219, T-87	Ti 6Al-4V, STA
CRES 304L, Annealed	1010 Steel, Normalized
CRES 347, Annealed	Cu OFHC, Annealed
CRES 17-4 PH, H-1025	Polytetrafluoroethylene
Inconel 718, STA	Kel F 81 CTFE
Monel 400, Annealed	Carbon CJPS
Nickel 200, Annealed	Kalrez, Epoxy EA 934

2.5.1 Apparatus and Procedures

2.5.1.1 Description of Apparatus

The requirements of the adiabatic compression test necessitates the use of an apparatus in which gaseous nitrogen trifluoride at variable initial temperatures and/or pressures is adiabatically compressed to variable compression ratios in the presence of selected material specimens. The apparatus previously used successfully under Contract F04611-72-C-0031 for similar adiabatic compression tests with fluorine and chlorine pentafluoride is employed for these tests with nitrogen trifluoride.

The apparatus is a U-tube adiabatic compression test apparatus which is modified to accommodate the introduction of gaseous nitrogen trifluoroide and material specimens and to incorporate a means of temperature conditioning the loaded U-tube. A schematic diagram of the entire apparatus for handling the nitrogen trifluoride and conducting the tests is shown in Figure 2.5.1. The schematic diagram of the U-tube adiabatic compression apparatus is shown in Figure 2.5.2; a photograph of the apparatus is shown in Figure 2.5.3; and the schematic of the test specimen holder with the test specimen in place is shown in Figure 2.5.4. The test specimen holder is a 0.64 cm (0.25 in.) solid AN plug used to seal the end of the U-tube. The test specimen is a strip of material 0.25 mm (0.010-in.) thick by 2.5 mm (0.10-in.) wide by 2.5 mm (0.10-in.) long which is spot welded, if metal, to the end of the AN plug or wedged into the end of a hollow AN plug and cemented in place with Sauereisen if the specimen is a non-metal. The U-tube is fabricated from Hastelloy-X 0.64 cm (0.25 in.) tubing approximately 40.6 cm (16-in.) long.



LEGEND:

BD	: BURST DISC	PG	: PRESSURE GAUGE
CV	: CHECK VALVE	R-1	: GASEOUS NF_3 REGULATOR
GN_2-A	: GASEOUS NITROGEN ACCUMULATOR	ROV	: REMOTE OPERATION VALVE (RUN VALVE)
GNF_3-C	: GASEOUS NF_3 CYLINDER	TRAP	: VACUUM TRAP
GNF_3-R	: GASEOUS NF_3 RESERVOIR	V-	: HAND OPERATED VALVE, 1/4-IN. CONTROL COMPONENTS

Figure 2.5.1. Schematic Diagram of System for Conducting the Adiabatic Compression Testing

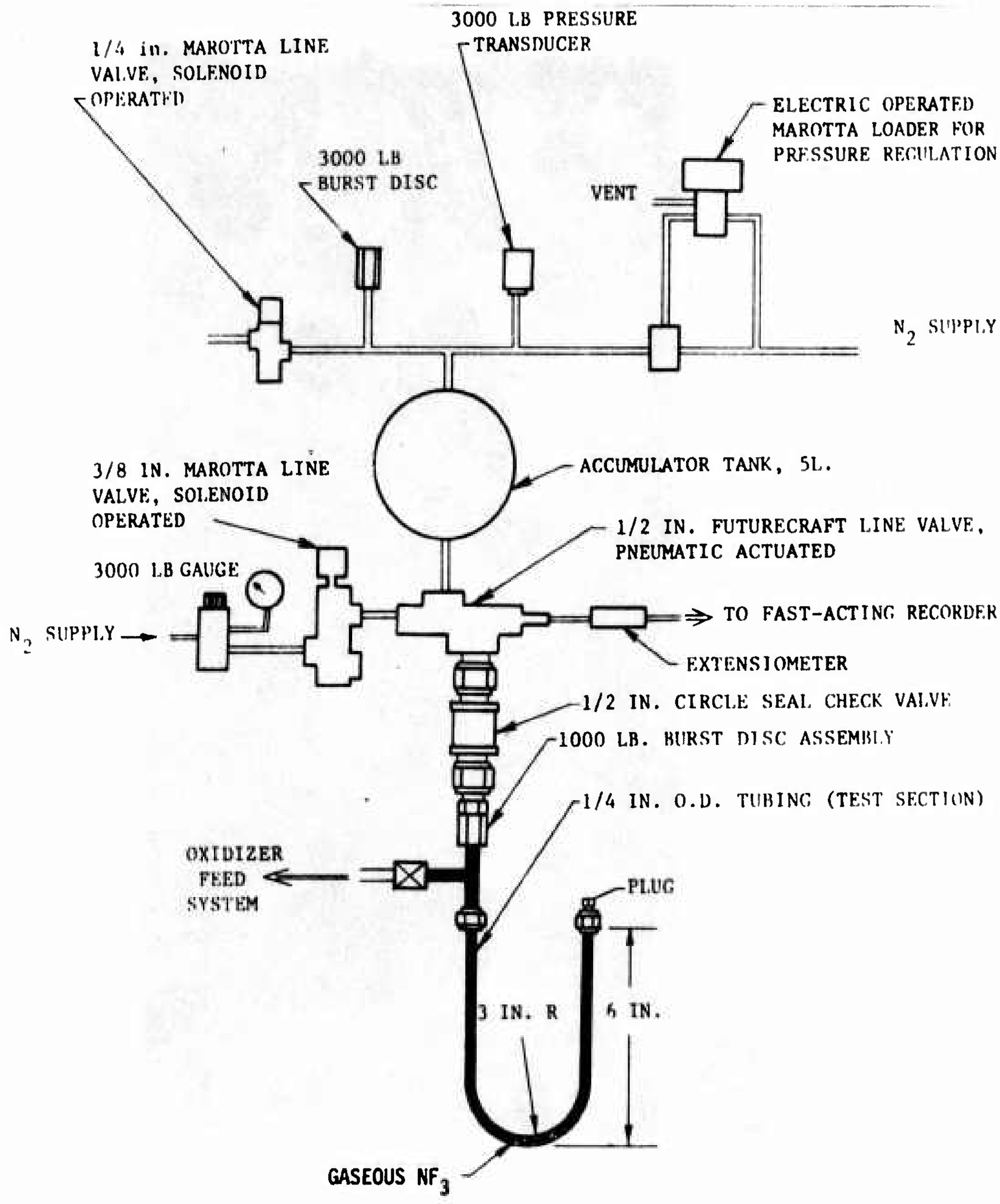


Figure 2.5.2. Schematic Diagram of U-tube Adiabatic Compression Apparatus

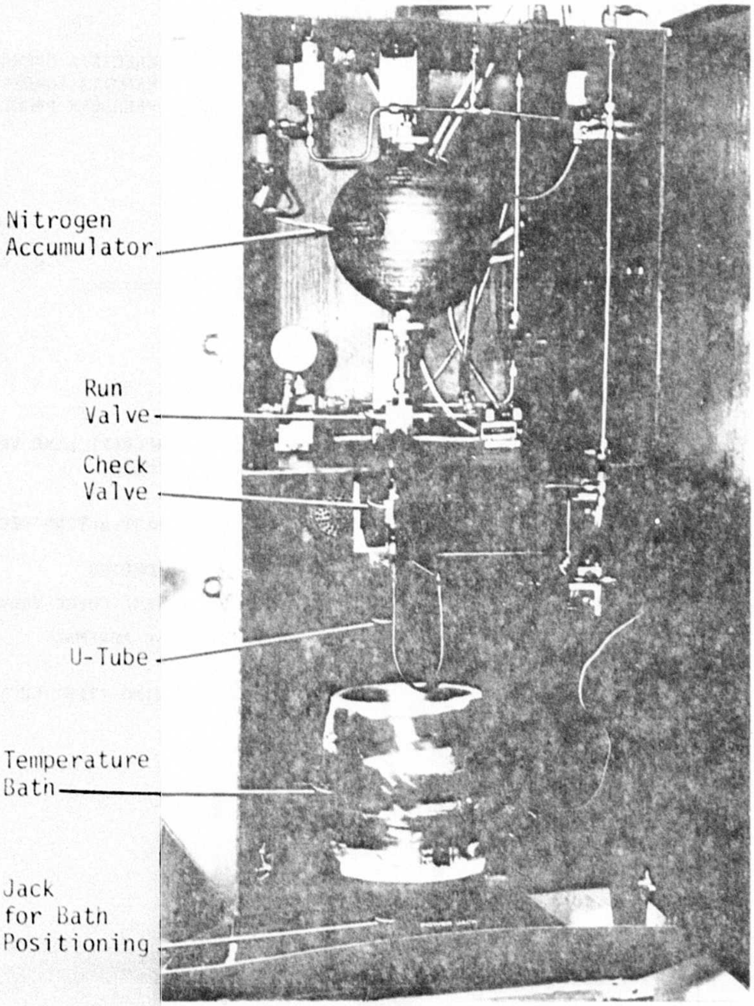


Figure 2.5.3. Adiabatic Compression Apparatus

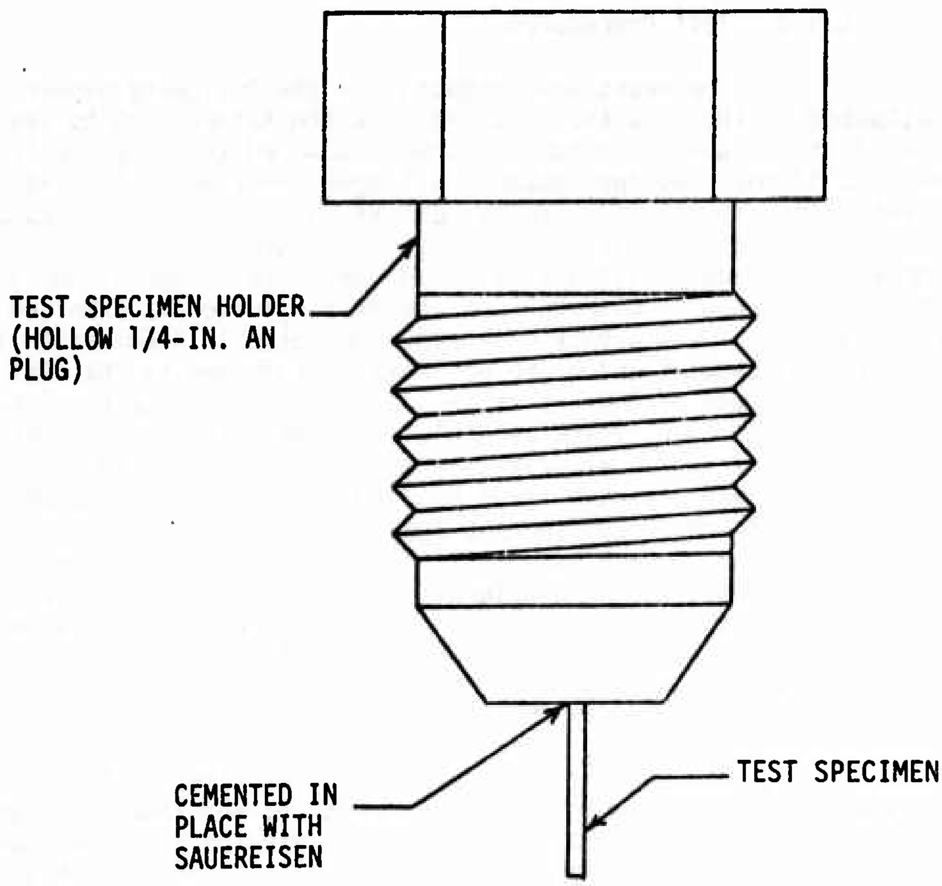


Figure 2.5.4. Schematic of Test Specimen Holder with Test Specimen in Place

2.5.1.2 Test Procedures

The tests are conducted in the following manner. The U-tube is attached to the apparatus and the specimen holder used to seal the open-end of the U-tube. The tube is then evacuated to 1 torr or less, temperature conditioned, and then gaseous nitrogen trifluoride is gradually introduced into the assembly to a predefined pressure level. The pneumatic line valves are then actuated and the nitrogen from the accumulator tank is used to compress the gaseous nitrogen trifluoride. The U-tube assembly is then vented and flushed with nitrogen and the test specimen is examined visually to ascertain if any attack occurred. Microscopic examination is used to evaluate the samples which are not totally consumed in the test. A 1000 lb burst disc made of 304-L stainless is used in each test to seal the pneumatic valve and check valve assembly from the nitrogen trifluoride atmosphere prior to the test. At driving pressures below 7.68 MN/m^2 (1100 psig) no burst disc was used. The driving pressure in the accumulator tank and the initial nitrogen trifluoride temperature and/or pressure is varied with each material to achieve final pressures and temperatures at which the metals are susceptible to attack. The test specimen is replaced after each test to insure that comparable surfaces are being exposed to the test conditions. The pneumatic valve opens completely withing 1.5 milliseconds so with an accumulator pressure of 6.89 MN/m^2 (1000 psia), the minimum pressurization rate is 4.6×10^6 $MN/m^2/sec$ (6.7×10^5 psi/sec).

The data obtained from the test directly includes the initial nitrogen trifluoride temperature and pressure, the final nitrogen trifluoride pressure (after adiabatic compression) and the test specimen response. The test specimen response is reported as (-) for a negative result indicating no microscopically visible effect, as (+) when the specimen shows definite attack but is not destroyed, and as (++) when the specimen is destroyed. Tests were conducted with both pure nitrogen trifluoride and a mixture of 15% nitrogen trifluoride - 85% argon which permitted significantly higher temperature levels to be attained and also significantly lower values for the density of the gaseous nitrogen trifluoride.

2.5.1.3 Calculation Procedures

The final temperature values were obtained from graphs of final pressure values versus temperature. The graphs were generated from entropy diagrams for gaseous nitrogen trifluoride and the gaseous nitrogen trifluoride/argon mixture.

2.5.1.3.1 Entropy of Gaseous Nitrogen Trifluoride

The entropy of ideal gaseous nitrogen trifluoride at one atmosphere pressure (0.101325 MN/m^2) and various temperatures was taken from the JANNAF Thermochemical Tables (Reference 2.5.1). The entropy of the real gas at various pressures and temperatures was determined by calculating isothermal entropy changes between real-gas and ideal-gas states and between pressures in the real-gas region and then applying those entropy changes to the ideal-gas data. The entropy changes are analytically given by equation (1) which is taken from Reference 2.5.2, p. 269.

$$S_2 - S_1 = \frac{1}{T_r}\left[\left(\frac{H_2 - H^o}{T_c}\right)_{Pr_2} - \left(\frac{H_1 - H^o}{T_c}\right)_{Pr_1}\right] - R\left[\ln\frac{f_2}{P_2} - \ln\frac{f_1}{P_1} + \ln\frac{P_2}{P_1}\right] \quad (1)$$

Values for the enthalpy deviations, $(H-H_o)/T_c$, and fugacity coefficients, f/P, were taken from Appendix B of Reference 2.5.2 at selected reduced temperatures, $T_r = T/T_c$, and reduced pressures, $P_r = P/P_c$, using values for the critical temperature and pressure, T_c and P_c, of nitrogen trifluoride from Jarry and Miller (Reference 2.5.3). The resulting entropy data for nitrogen trifluoride are given in Table 2.5-1, and are presented in the form of a T-S diagram in Figure 2.5.5.

To aid in the selection of test conditions for the adiabatic compression tests and in the reduction of experimental test data, the entropy data from Table 2.5-1 were interpolated to yield curves relating nitrogen trifluoride temperatures following adiabatic compression from fixed initial pressures and various initial temperatures to various final pressures. In Figures 2.5.6 and 2.5.7 the initial nitrogen trifluoride pressure is 34.47 KN/m^2 (5 psia) and curves are given for initial nitrogen trifluoride temperatures in the range of 283.16 to 298.16 K (10 to 25 C) and final pressures in the ranges of 0.2758 to 2.758 MN/m^2 (40 to 400 psia) and 2.758 to 20.684 MN/m^2 (400 to 3000 psia), respectively. Similar curves are shown in Figures 2.5.8 and 2.5.9 except the initial nitrogen trifluoride pressure in these cases is 0.1013 MN/m^2 (1 atm).

2.5.1.3.2 Entropy of Gaseous Nitrogen Trifluoride-Argon Mixtures

The entropies of nitrogen trifluoride-argon mixtures were calculated assuming the ideal mixing of the components starting with each component at the pressure of the mixture and treating each component as a real-gas. In such cases the entropy of the mixture is defined analytically by Equation (2):

TABLE 2.5-1

ENTROPY OF NF_3

Press., MN/m^2:		Entropy, J/mol·K											
		0.03447	0.06895	0.1013	0.1379	0.2758	0.4137	0.6895	1.379	2.758	6.895	13.790	20.684
psia :		5	10	14.696	20	40	60	100	200	400	1000	2000	3000
Temperature K	F												
200	-99.69	250.53	244.76	241.42	238.72	232.6	228.9	223.8	215.8	--	--	--	--
300	80.31	269.96	264.19	260.96	258.37	252.54	249.09	244.67	238.46	231.68	220.04	208.51	203.35
400	260.31	286.57	280.80	277.60	275.02	269.24	265.85	261.55	255.64	249.52	240.56	233.00	228.46
500	440.31	301.03	295.26	292.06	289.50	283.72	280.33	276.06	270.23	264.30	256.03	249.28	245.12
600	620.31	313.71	307.95	304.75	302.19	296.42	293.03	288.77	282.96	277.04	269.09	262.83	259.07
700	800.31	324.93	319.16	315.96	313.40	307.63	304.26	300.00	294.21	288.38	280.51	274.55	271.04
800	980.31	334.96	329.19	325.99	323.43	317.67	314.29	310.04	304.26	298.46	290.72	284.79	281.32
900	1160.31	343.99	338.22	335.02	332.46	326.70	323.32	319.08	313.30	307.52	299.83	293.96	290.51
1000	1340.31	352.19	346.43	343.23	340.67	334.90	331.53	327.28	321.51	315.73	308.07	302.24	298.81
1100	1520.31	359.70	353.93	350.73	348.17	342.41	339.03	334.79	329.03	323.25	315.60	309.79	306.38
1200	1700.31	366.61	360.84	357.64	355.08	349.32	345.95	341.70	335.94	330.18	322.53	316.74	313.34
1300	1880.31	373.01	367.25	364.05	361.49	355.72	352.35	348.10	342.34	336.58	328.95	323.16	319.78
1400	2060.31	378.97	373.21	370.01	367.45	361.68	358.31	354.06	348.30	342.54	334.92	329.14	325.73
1500	2240.31	384.55	378.78	375.58	373.02	367.25	363.88	359.64	353.87	348.11	340.49	334.73	331.36
1600	2420.31	389.78	384.01	380.81	378.25	372.48	369.11	364.87	359.10	353.34	345.72	339.96	336.59
1700	2600.31	394.71	388.94	385.74	383.18	377.41	374.04	369.79	364.03	358.27	350.65	344.89	341.52
1800	2780.31	399.36	393.60	390.40	387.84	382.07	378.70	374.45	368.69	362.93	355.31	349.54	346.18
1900	2960.31	403.78	398.02	394.81	392.25	386.49	383.12	378.87	373.11	367.35	359.73	353.96	350.59
2000	3140.31	407.98	402.21	399.01	396.45	390.69	387.31	383.07	377.30	371.54	363.92	358.16	354.79

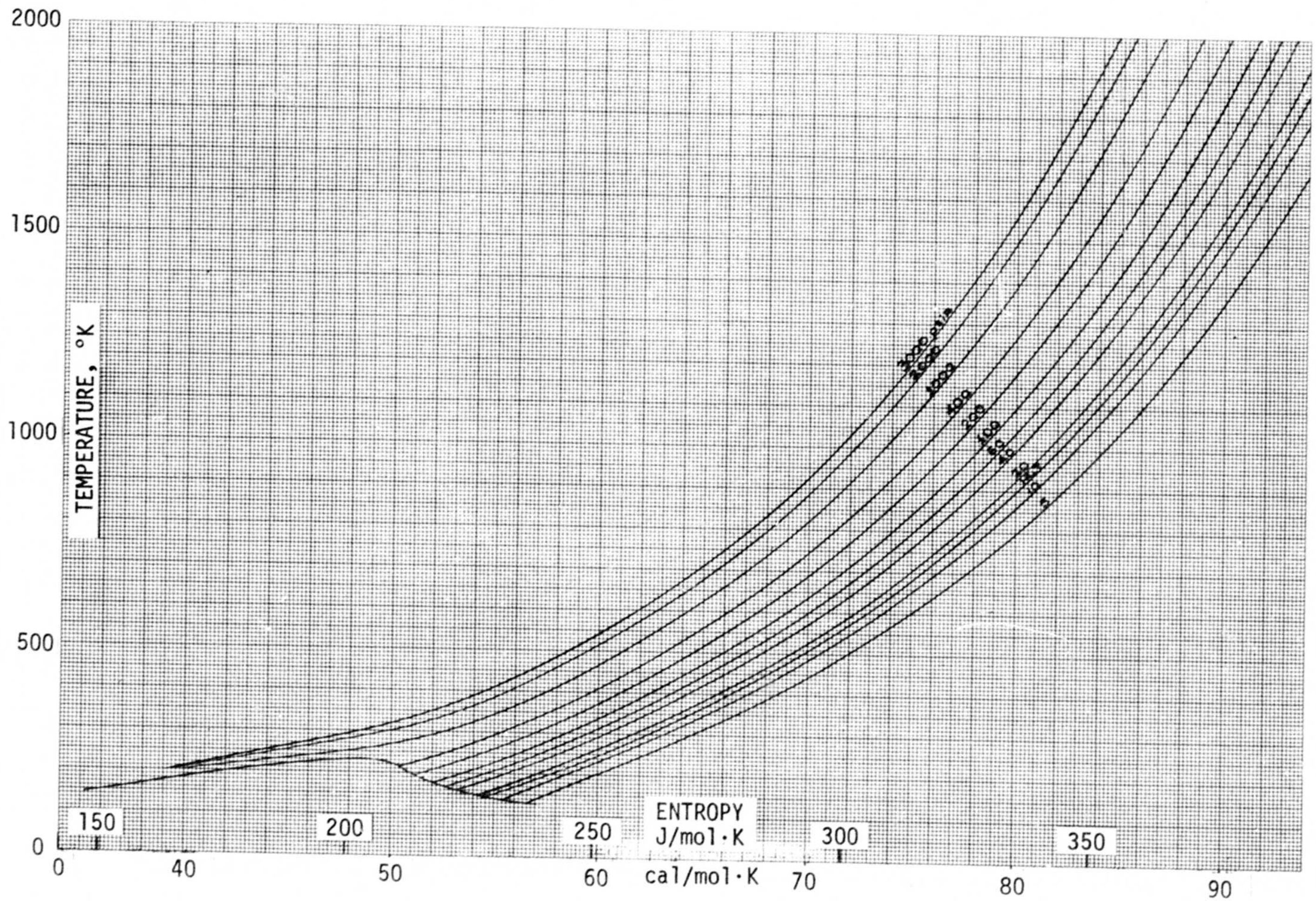


Figure 2.5.5. Temperature-Entropy Diagram for Nitrogen Trifluoride

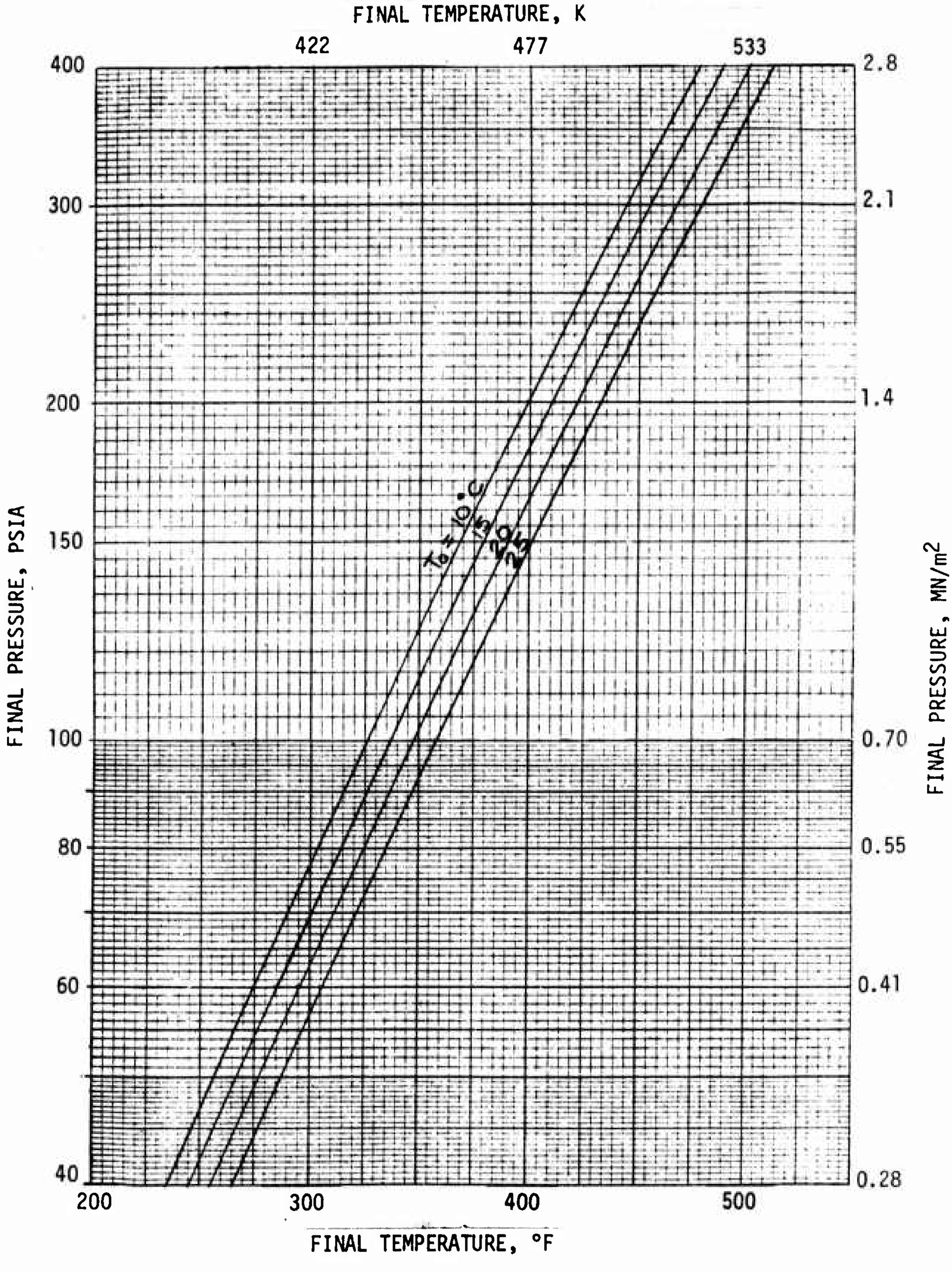


Figure 2.5.6. Final NF_3 Temperatures Resulting from Adiabatic Compression from an Initial Pressure of 34.47 KN/m^2 (5 psia) to Final Pressures in the Range of 0.2758-2.758 MN/m^2 (40-400 psia)

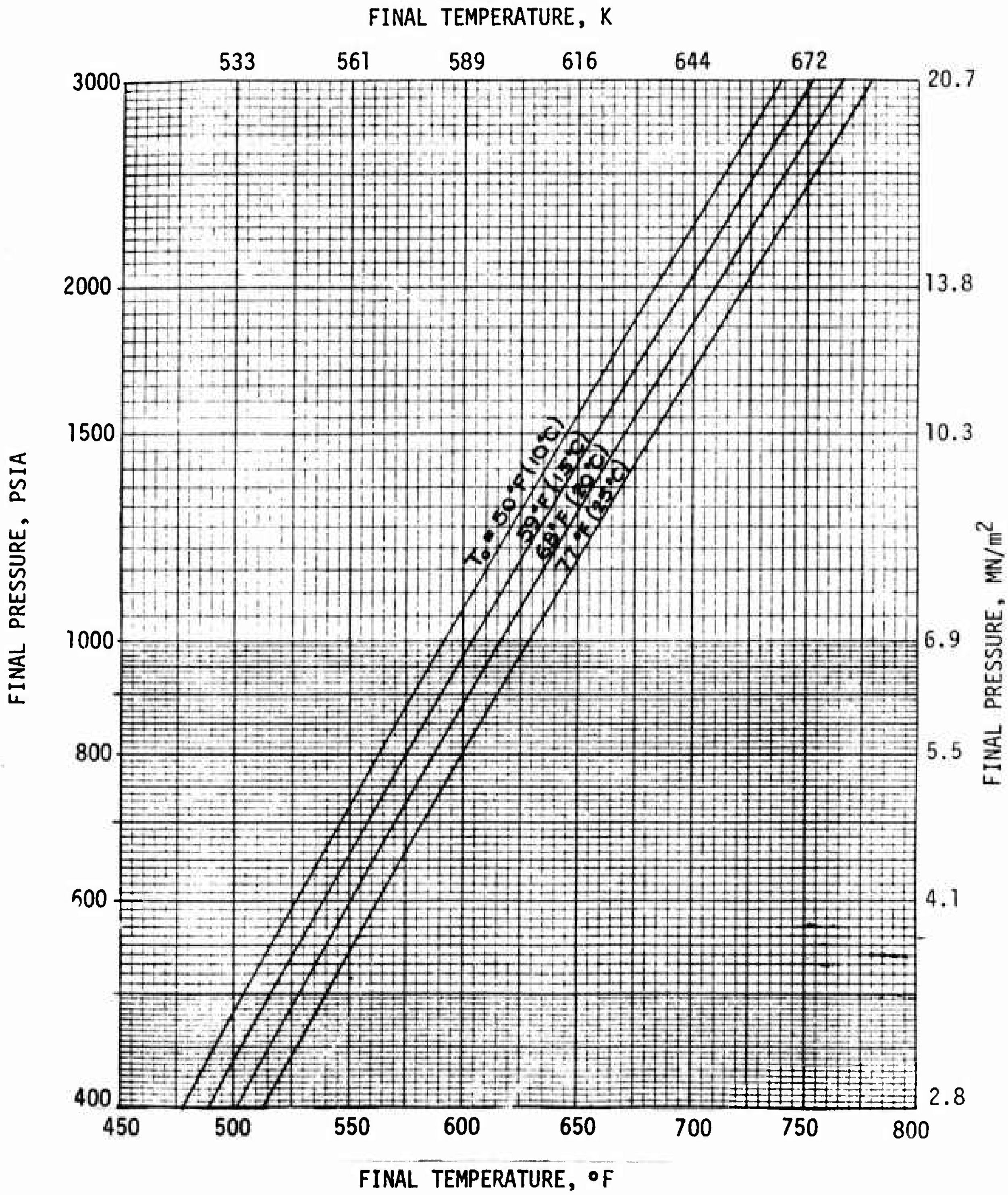


Figure 2.5.7. Final NF_3 Temperatures Resulting from Adiabatic Compression from an Initial Pressure of 34.47 KN/m^2 (5 psia) to Final Pressures in the Range of 2.758-20.684 MN/m^2 (400-3000 psia)

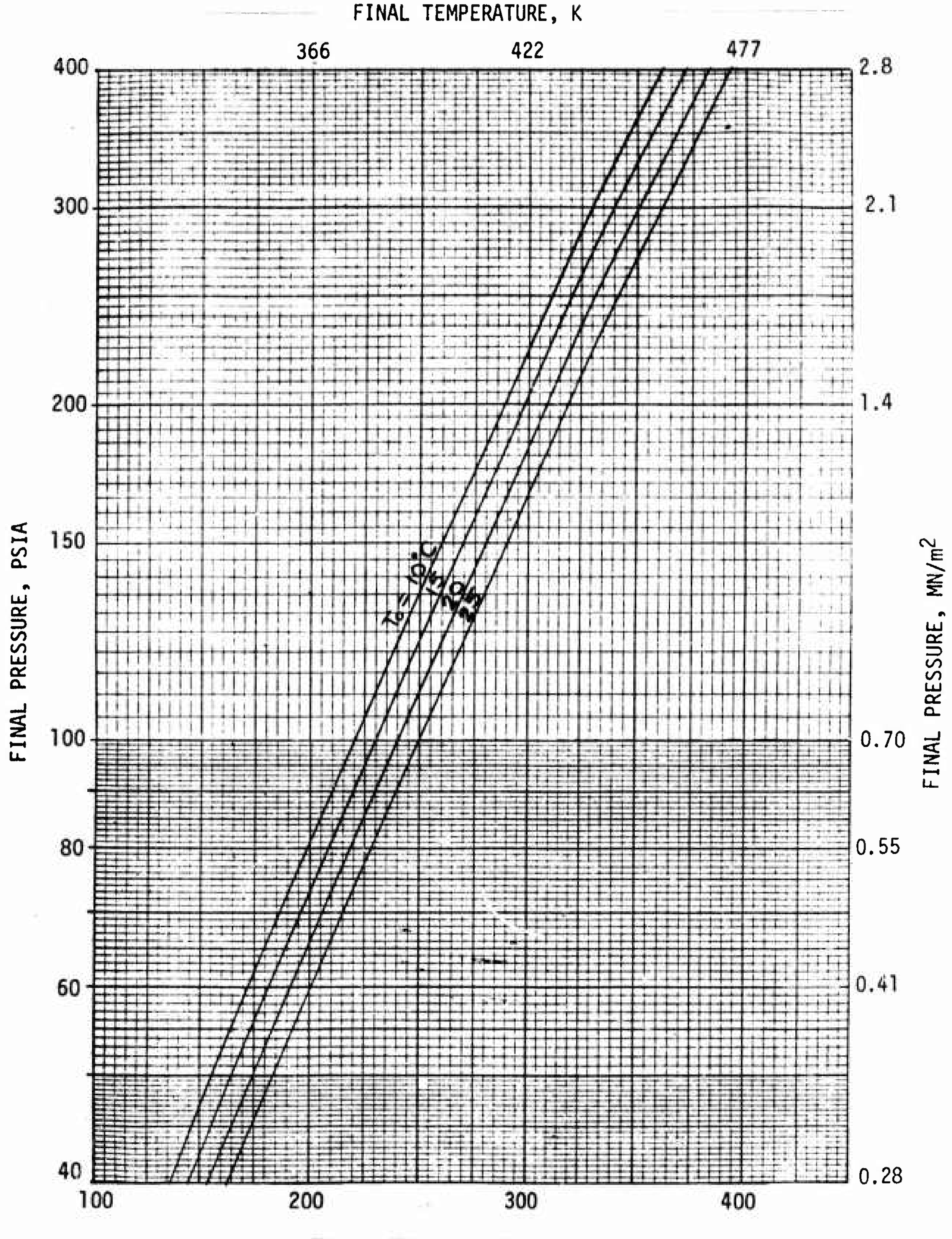


Figure 2.5.8. Final NF_3 Temperatures Resulting from Adiabatic Compression from an Initial Pressure of 0.1013 MN/m^2 (1 atm) to Final Pressures in the Range of 0.2758-2.758 MN/m^2 (40-400 psia)

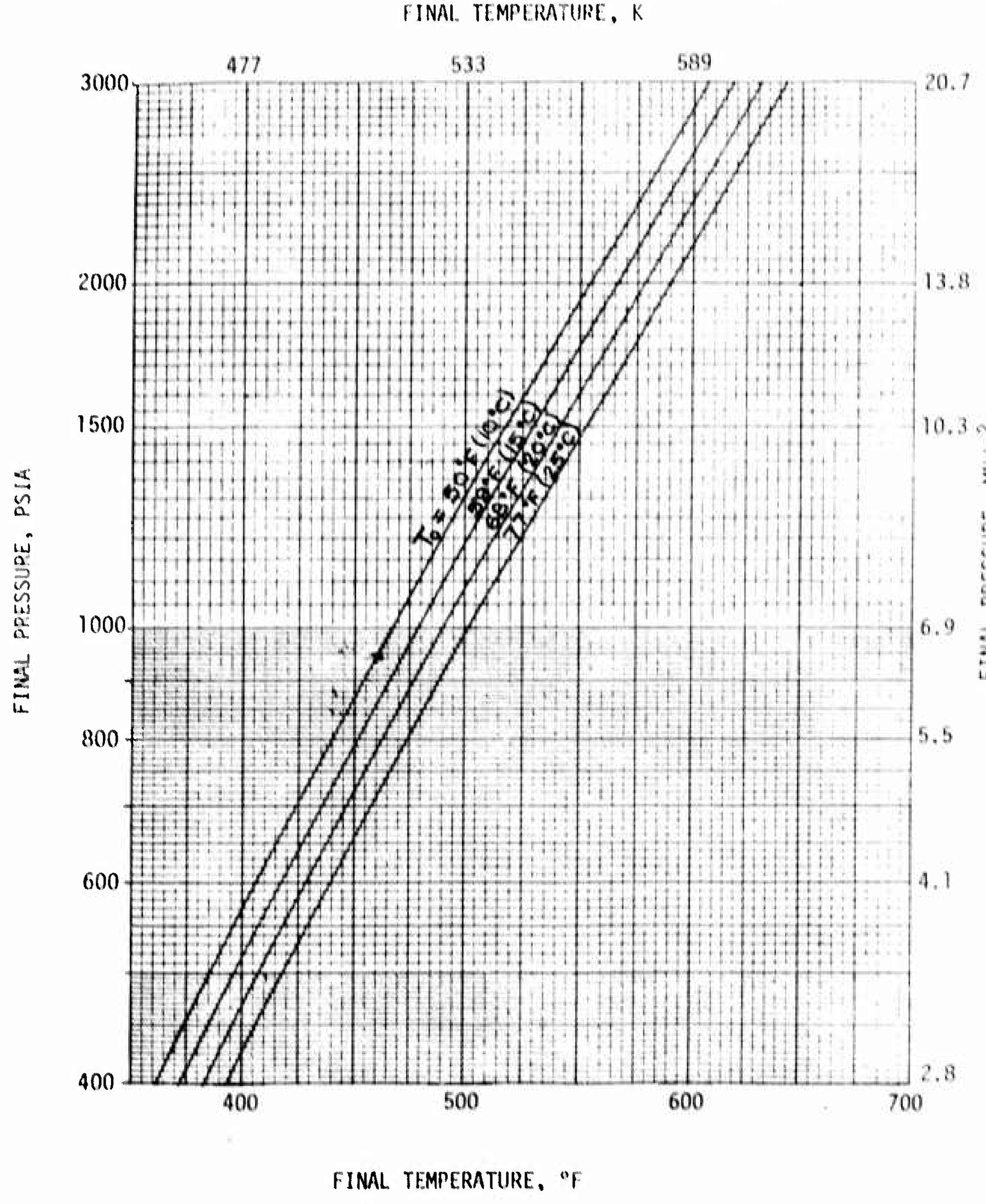


Figure 2.5.9. Final NF_3 Temperatures Resulting from Adiabatic Compression from an Initial Pressure of 0.1013 MN/m^2 (1 atm) to Final Pressures in the Range of 2.758-20.684 MN/m^2 (400-3000 psia)

$$S_{mix} = \sum y_i S_i - R\sum y_i \ln y_i \qquad (2)$$

where:

S_i = the entropy of the ith component at the pressure and temperature of the mixture, and

y_i = mole fraction of the ith component in the mixture.

In these calculations, values for the entropy of nitrogen trifluoride (real-gas) were taken from Table 2.5.1 and for argon (real-gas) from Reference 2.5.4. For argon, real-gas entropies at pressures above 10.1325 MN/m^2 (100 atm) were obtained by logarithmic extrapolation of values given at 70 and 100 atm. Argon entropies at pressures between 0.01 and 100 atm were obtained by logarithmic interpolation of the tables given in Reference 2.5.4.

On the basis of the calculated temperature-entropy data for nitrogen trifluoride-argon mixtures containing 10, 15, 20, 25, and 50% vol nitrogen trifluoride, a mixture containing 15% vol nitrogen trifluoride was selected as being most useful in adiabatic compression tests aimed at defining threshold material compatibility limits with nitrogen trifluoride in the high temperature-low density regime. As a further aid in the selection of adiabatic compression test conditions and in the reduction of experimental test data, the temperature-entropy data for nitrogen trifluoride-argon (15/85) were interpolated to yield curves relating nitrogen trifluoride temperatures following adiabatic compression to pressures in the rangeof 2.758 to 20.684 MN/m^2 (400 to 3000 psia) wherein the initial pressure is 0.1013 MN/m^2 (1 atm) and initial temperatures are in the range of 283.16 to 298.16 K (10 to 25 C). These curves are presented in Figure 2.5.10.

2.5.1.3.3 Final Density Calculation

The final gas density value for the tests in which pure nitrogen trifluoride was used was calculated on the basis of the gas law:

$$\rho = \frac{(MW)(P)}{ZRT}$$

The compressibility factor (Z) was assumed to be unity.

In the tests in which the gaseous mixture of 15% NF_3-85% Ar (volume percents) was used, the final gas density was calculated from the gas law equation using a mean molecular weight value of 44.61 for the mixture and then multiplying the calculated density value by the weight fraction of nitrogen trifluoride in the mixture, 0.2388, to obtain the nitrogen trifluoride density values which are reported in Table 2.5-2.

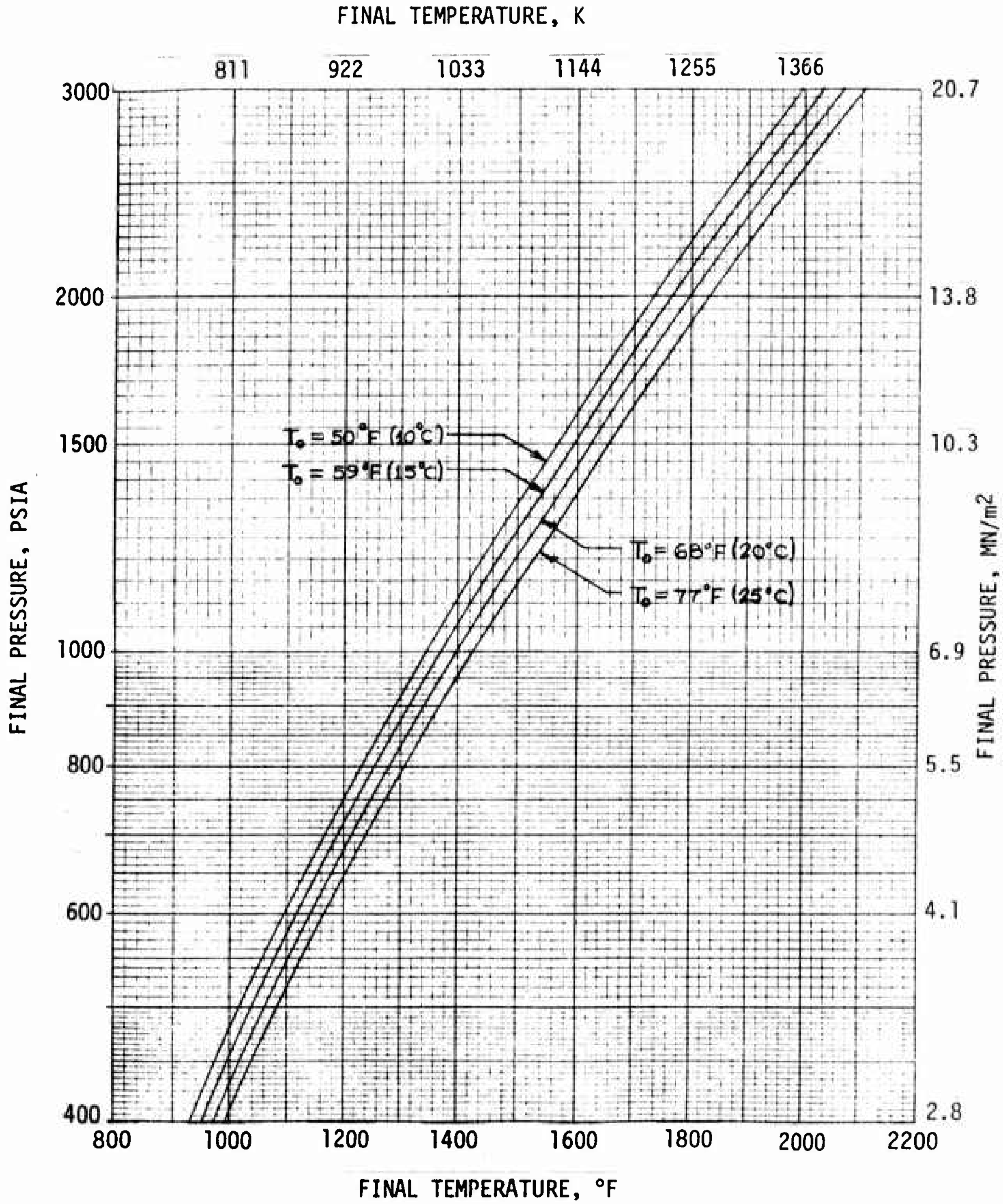


Figure 2.5.10. Final NF_3-Ar (15/85) Temperatures Resulting from Adiabatic Compression from an Initial Pressure of 0.1013 MN/m^2 (1 atm) to Final Pressures in the Range of 2.758-20.684 MN/m^2 (400-3000 psia)

2.5.2 Experimental Results

The data which were derived from the adiabatic compression tests are presented in Table 2.5-2. The initial pressure values which are presented in paretheses are for the 15% nitrogen trifluoride-85% argon mixture and the pressure value corresponds to the partial pressure of the nitrogen trifluoride present in the mixture. The data in Table 2.5-2 are presented in both SI units and in English units. The test results are based on the visual microscopic observations prior to and after each test. In many cases the decreasing severity of the attack was noted as the driving pressures were decreased. The surface changes observed for the metal specimens were generally minor and might fall into a category of being comparable to the formation of passivation films. None of the metal samples appeared to react in a manner which would result in the ignition of the material.

In order to obtain additional information on the nature of the reaction which occurs with the metal surfaces, a Ni 200 sample was subjected repeatedly to the adiabatic compression test and the results are presented in Table 2.5-3. The data indicate that additional surface reaction occurs with each adiabatic compression cycle above the threshold level defined in Table 2.5-2, and implies that the reaction which occurs is more severe than one would anticipate from a passivation reaction, i.e., pitting occurs.

The threshold conditions for each material as identified by the adiabatic compression tests are summarized in Table 2.5-4. The data in the table corresponds to the final conditions at which no reaction was observed.

Although the adiabatic compression environment is complex due to the shock waves which are also present during the process and this complicates the interpretation of the data for design purposes, there are a significant number of items to note from the data which is given in Table 2.5-2 and summarized in Table 2.5-4. The items to note are as follows: (1) definite threshold limits exist for the various materials when they are subjected to adiabatic compression of nitrogen trifluoride from various initial conditions and these thresholds provide a basis for rating the relative resistance of materials to nitrogen trifluoride attack during compression; (2) of the metals tested, 304L stainless steel and Nickel-200 rate best while carbon steel, copper, and titanium rate poorly; (3) of the non-metals tested, carbon CJPS rates best and is comparable to many of the intermediate metals; and (4) some of the best fluorocarbon plastics such as polytetrafluoroethylene, Kel-F 81, and Kalrez exhibit moderate compatibility but comparable only to some of the less compatible metals.

TABLE 2.5-2

DATA INDICATIVE OF THE BEHAVIOR OF MATERIALS IN THE PRESENCE OF GASEOUS NITROGEN TRIFLUORIDE SUBJECTED TO ADIABATIC COMPRESSION

Material	Initial Condition				Final Condition						Test Results
	Temperature		Pressure		Pressure		Calculated Temperature		Calculated Density		
	°K	°F	kN/m2	psia	MN/m2	psia	K	F	kg/m3	lb/ft3	
Aluminum 2219 T-87	292	66	101	14.7	11.8	1715	564	556	175.7	10.97	+, Some surface change
	292	66	101	14.7	10.4	1515	556	540	160.8	10.04	+, Some surface change
	292	66	101	14.7	9.76	1415	551	532	151.5	9.46	-
	292	66	101	14.7	8.38	1215	540	512	132.8	8.29	-
	292	66	33.8	4.9	11.8	1715	637	687	158.9	9.92	+, Considerable surface change
	292	65	33.8	4.9	3.55	515	550	530	55.3	3.45	+, Slight surface change
	293	68	33.8	4.9	2.86	415	536	504	45.7	2.85	+, Slight surface change
	293	68	33.8	4.9	2.17	315	517	470	36.0	2.25	-
	295	71	15.2	(2.2)	13.9	2015	1255	1800	14.3	0.89	+, Slight surface change
	295	71	15.2	(2.2)	12.5	1815	1222	1740	13.1	0.82	+, Slight surface change
	295	71	15.2	(2.2)	11.8	1715	1203	1705	12.7	0.79	+, Very slight surface change
	295	71	15.2	(2.2)	11.1	1615	1183	1670	12.0	0.75	-
Stainless Steel 304L, annealed	292	66	101	14.7	13.9	2015	576	577	206.2	12.88	-
	292	66	33.8	4.9	13.9	2015	649	708	183.1	11.44	-
	295	71	15.2	(2.2)	13.9	2015	1255	1800	14.3	0.89	-
	295	71	15.2	(2.2)	13.9	2015	1255	1800	14.3	0.89	-
Stainless Steel 347, annealed	290	62	101	14.7	13.9	2015	572	570	207.8	12.97	+, Very slight surface change
	291	64	101	14.7	13.2	1915	570	567	198.1	12.37	-
	291	64	33.8	4.9	4.93	715	570	567	74.0	4.62	+, Some surface change
	291	64	33.8	4.9	4.24	615	560	548	64.7	4.04	-
	291	64	33.8	4.9	3.55	515	546	523	55.6	3.47	-
	293	67	15.2	(2.2)	13.9	2015	1255	1800	14.3	0.89	+, Slight surface change
	287	57	15.2	(2.2)	13.2	1915	1222	1740	12.1	0.87	+, Very slight surface change
	287	57	15.2	(2.2)	12.5	1815	1200	1700	13.5	0.84	-
	293	67	15.2	(2.2)	11.8	1715	1203	1705	12.7	0.79	-
	293	67	15.2	(2.2)	10.4	1515	1161	1630	11.5	0.72	-

TABLE 2.5-2 (cont.)

Material	Initial Condition Temperature °K	Initial Condition Temperature °F	Initial Condition Pressure kN/m2	Initial Condition Pressure psia	Final Condition Pressure MN/m2	Final Condition Pressure psia	Calculated Temperature K	Calculated Temperature F	Calculated Density kg/m3	Calculated Density lb/ft3	Test Results
Stainless Steel 17-4PH, H-1025	294	69	101	14.7	12.5	1815	570	567	187.9	11.73	+, Slight surface change
	294	69	101	14.7	11.1	1615	562	551	169.5	10.58	+, Slight surface change
	294	69	101	14.7	10.4	1515	557	542	160.5	10.02	-
	286	55	33.8	4.9	4.24	615	552	533	65.7	4.10	+, Some surface change
	286	55	33.8	4.9	2.86	415	525	486	46.6	2.91	+, Some surface change
	286	55	33.8	4.9	1.48	215	484	412	26.3	1.64	+, Very slight surface change
	286	55	33.8	4.9	1.14	165	469	385	20.7	1.29	-
	287	57	33.8	4.9	0.79	115	447	345	15.2	0.95	-
	293	67	15.2	(2.2)	13.9	2015	1255	1800	14.3	0.89	+, Slight surface change
	294	69	15.2	(2.2)	12.5	1815	1222	1740	13.1	0.82	+, Surface change
	295	71	15.2	(2.2)	11.8	1715	1205	1710	12.7	0.79	-
	295	71	15.2	(2.2)	11.1	1615	1189	1680	12.0	0.75	-
	294	69	15.2	(2.2)	9.76	1415	1139	1590	11.1	0.69	-
Inconel 718, STA	294	69	101	14.7	13.9	2015	578	580	205.7	12.84	+, Slight surface change
	294	69	101	14.7	12.5	1815	570	566	187.9	11.73	+, Some pitting of the surface
	294	69	101	14.7	10.4	1515	556	542	160.8	10.04	+, Slight surface change
	294	69	101	14.7	8.38	1215	542	515	132.3	8.26	+, Some surface change
	294	69	101	14.7	7.00	1015	529	492	113.3	7.07	+, Some surface change
	294	69	101	14.7	6.31	915	525	485	102.8	6.42	+, Slight surface change
	294	69	101	14.7	5.62	815	514	465	93.5	5.84	-
	294	69	33.8	4.9	3.55	515	551	532	55.1	3.44	+, Some surface change
	294	69	33.8	4.9	2.86	415	536	506	45.7	2.85	-
	294	69	33.8	4.9	2.17	315	518	473	35.9	2.24	-
	295	71	15.2	(2.2)	13.9	2015	1264	1815	14.1	0.88	+, Slight surface change
	295	71	15.2	(2.2)	13.2	1915	1244	1780	13.6	0.85	-
Monel 400, Annealed	289	60	101	14.7	13.9	2015	570	567	208.6	13.02	+, Very slight surface change
	289	60	101	14.7	13.2	1915	566	560	199.6	12.46	+, Slight surface change
	289	60	101	14.7	11.1	1615	555	539	171.7	10.72	+, Slight surface change
	290	62	101	14.7	8.38	1215	534	502	134.2	8.38	+, Slight surface change
	291	64	101	14.7	7.69	1115	532	497	123.7	7.72	+, Slight surface change
	290	62	101	14.7	7.00	1015	524	483	114.2	7.13	-
	291	64	33.8	4.9	2.86	415	533	500	46.0	2.87	+, Slight surface change
	291	64	33.8	4.9	2.17	315	515	467	36.0	2.25	+, Slight surface change
	291	64	33.8	4.9	1.48	215	490	423	25.9	1.62	-
	293	68	15.2	(2.2)	13.9	2015	1255	1800	14.3	0.89	-

TABLE 2.5-2 (cont.)

Material	Initial Condition Temperature °K	°F	Pressure kN/m²	psia	Final Condition Pressure MN/m²	psia	Calculated Temperature K	F	Calculated Density kg/m3	lb/ft3	Test Results
Nickel 200, Annealed	292	66	101	14.7	13.9	2015	573	572	207.4	12.95	+, Slight surface change
	292	66	101	14.7	13.2	1915	572	570	197.5	12.33	-
	292	66	33.8	4.9	13.2	1915	645	702	175.1	10.93	+, Slight surface change
	292	66	33.8	4.9	12.5	1815	642	695	166.8	10.41	+, Slight surface change
	292	66	33.8	4.9	11.8	1715	636	686	159.1	9.93	+, Slight surface change
	292	66	33.8	4.9	10.4	1515	628	670	142.4	8.89	+, Some surface change
	292	66	33.8	4.9	9.07	1315	616	650	125.9	7.86	+, Some surface change
	292	66	33.8	4.9	8.38	1215	610	639	117.6	7.34	-
	292	66	33.8	4.9	7.69	1115	604	628	108.6	6.78	-
	298	76	15.2	(2.2)	13.9	2015	1272	1830	14.1	0.88	-
Titanium 6Al-4V, STA	286	55	101	14.7	6.31	915	512	462	105.4	6.58	+, Some surface change
	286	55	101	14.7	3.55	515	475	395	63.9	3.99	+, Some surface change
	286	55	101	14.7	2.86	415	460	368	53.2	3.32	-
	287	57	101	14.7	2.17	315	447	345	41.6	2.60	-
	287	57	33.8	4.9	0.79	115	450	350	15.1	0.94	+, Slight surface change
	286	55	33.8	4.9	.45	65	415	287	9.3	0.58	-
	294	69	15.2	(2.2)	13.9	2015	1255	1800	14.3	0.89	+, Surface change
	294	69	15.2	(2.2)	12.5	1815	1222	1740	13.1	0.82	+, Some surface change
	294	69	15.2	(2.2)	11.1	1615	1183	1670	12.0	0.75	+, Slight surface change
	294	69	15.2	(2.2)	9.76	1415	1139	1590	11.1	0.69	+, Slight surface change
	294	69	15.2	(2.2)	8.38	1215	1089	1500	9.9	0.62	+, Slight surface change
	294	69	15.2	(2.2)	7.00	1015	1036	1405	8.6	0.54	+, Slight surface change
	294	69	15.2	(2.2)	6.31	915	1005	1350	8.0	0.50	-
	294	69	15.2	(2.2)	5.62	815	972	1290	7.4	0.46	-
1010 Steel	291	64	101	14.7	13.9	2015	574	574	207.1	12.93	+, Surface change
	292	66	101	14.7	10.4	1515	555	540	161.0	10.05	+, Surface change with pitting
	292	66	101	14.7	9.07	1315	546	523	142.1	8.87	+, Some surface change
	292	66	101	14.7	7.69	1115	534	501	123.2	7.69	+, Some surface change
	292	66	101	14.7	7.00	1015	529	492	113.3	7.07	+, Very slight surface change
	292	66	101	14.7	6.31	915	519	475	104.0	6.49	+, Very slight surface change
	292	66	101	14.7	5.62	815	513	463	93.7	5.85	-
	292	66	33.8	4.9	2.17	315	516	470	36.0	2.25	+, Slight surface change
	293	68	33.8	4.9	1.48	215	494	430	25.6	1.60	+, Some surface change
	293	68	33.8	4.9	1.14	165	478	400	20.3	1.27	+, Some surface change

TABLE 2.5-2 (cont.)

Material	Initial Condition				Final Condition						Test Results
	Temperature		Pressure		Pressure		Calculated Temperature		Calculated Density		
	°K	°F	kN/m2	psia	MN/m2	psia	K	F	kg/m3	lb/ft3	
1010 Steel (Cont.)	293	68	33.8	4.9	0.79	115	457	363	14.9	0.93	-
	291	64	15.2	(2.2)	13.9	2015	1247	1785	14.3	0.89	+, Slight surface change
	291	64	15.2	(2.2)	10.4	1515	1150	1610	11.7	0.73	+, Slight surface change
	291	64	15.2	(2.2)	9.07	1315	1105	1530	10.6	0.66	+, Some surface change
	291	64	15.2	(2.2)	8.38	1215	1080	1485	9.9	0.62	+, Very slight surface change
	286	55	15.2	(2.2)	7.69	1115	1039	1410	9.5	0.59	+, Some surface change
	292	66	15.2	(2.2)	7.00	1015	1033	1400	8.6	0.54	-
Copper OFHC, Annealed	287	57	101	14.7	9.07	1315	540	512	143.7	8.97	+, Some surface change
	288	58	101	14.7	7.69	1115	529	492	124.3	7.76	+, Very slight surface change
	288	58	101	14.7	7.00	1015	522	480	114.7	7.16	-
	289	60	33.8	4.9	2.86	415	530	494	46.1	2.88	+, Slight surface change
	289	60	33.8	4.9	1.48	215	489	420	25.9	1.62	+, Surface change
	290	62	33.8	4.9	0.79	115	452	353	15.1	0.94	+, Surface change
	290	62	33.8	4.9	0.45	65	420	297	9.1	0.57	+, Surface change
	298	76	15.2	(2.2)	10.4	1515	1178	1660	11.4	0.71	+, Surface pitted
	298	76	15.2	(2.2)	9.76	1415	1155	1620	11.1	0.69	+, Surface pitted
	298	76	15.2	(2.2)	9.07	1315	1133	1580	10.3	0.64	+, Slightly pitted surface
	298	76	15.2	(2.2)	8.38	1215	1105	1530	9.8	0.61	-
Polytetra-fluoroethylene	297	75	101	14.7	13.9	2015	583	590	203.9	12.73	++, Char spots, surface removed
	297	75	101	14.7	5.62	815	519	475	92.6	5.78	+, Slight char, slight surface change
	297	75	101	14.7	4.93	715	510	458	82.7	5.16	-
	297	75	101	14.7	3.55	515	489	420	62.2	3.88	-
	297	75	33.8	4.9	3.55	515	556	542	54.6	3.41	+, Slight surface change
	297	75	33.8	4.9	2.86	415	542	515	45.2	2.82	-
	297	75	33.8	4.9	2.17	315	523	482	35.6	2.22	-
	291	64	15.2	(2.2)	13.9	2015	1272	1830	14.1	0.88	+, Surface change, material removed
	291	64	15.2	(2.2)	12.5	1815	1233	1760	13.0	0.81	+, Some surface removed
	291	64	15.2	(2.2)	11.1	1615	1194	1690	12.0	0.75	+, Surface change
	291	64	15.2	(2.2)	10.4	1515	1175	1655	11.4	0.71	+, Surface change
	291	64	15.2	(2.2)	9.07	1315	1128	1570	10.3	0.64	+, Very slight surface change
	291	64	15.2	(2.2)	8.38	1215	1103	1525	9.8	0.61	-

TABLE 2.5-2 (cont.)

Material	Initial Condition				Final Condition						
	Temperature		Pressure		Pressure		Calculated Temperature		Calculated Density		
	°K	°F	kN/m2	psia	MN/m2	psia	K	F	kg/m3	lb/ft3	Test Results
Kel F 81 CTFE	287	57	101	14.7	9.76	1415	544	520	153.5	9.58	++, Specimen burned
	287	57	101	14.7	8.38	1215	533	500	131.8	8.23	++, Specimen burned
	287	57	101	14.7	7.69	1115	528	490	124.6	7.78	+, Slight surface change
	287	57	101	14.7	7.00	1015	521	478	115.0	7.18	-
	287	57	33.8	4.9	2.86	415	529	492	46.3	2.89	+, Very slight surface change
	287	57	33.8	4.9	2.17	315	508	455	36.5	2.28	-
	288	58	15.2	(2.2)	13.9	2015	1236	1765	14.4	0.90	+, Surface melting, char spot
	290	62	15.2	(2.2)	12.5	1815	1211	1720	13.3	0.83	+, Surface melting
	290	62	15.2	(2.2)	11.8	1715	1186	1675	12.8	0.80	+, Surface pitted and melted
	290	62	15.2	(2.2)	11.1	1615	1169	1645	12.2	0.76	+, Some surface melting
	290	62	15.2	(2.2)	10.4	1515	1150	1610	11.7	0.73	+, Very slight surface change
	288	58	15.2	(2.2)	10.4	1515	1144	1600	11.7	0.73	-
	290	62	15.2	(2.2)	9.76	1415	1125	1565	11.1	0.69	-
Carbon CJPS	294	69	101	14.7	13.9	2015	578	580	205.7	12.84	+, Some surface pitting
	294	69	101	14.7	12.5	1815	570	567	187.9	11.73	+, Some surface change
	294	69	101	14.7	11.8	1715	566	559	178.8	11.16	+, Very slight pitting
	294	69	101	14.7	11.1	1615	562	552	169.5	10.58	-
	293	67	33.8	4.9	4.93	715	573	572	73.7	4.60	+, Slight pitting
	293	67	33.8	4.9	4.24	615	562	552	64.6	4.03	-
	286	55	15.2	(2.2)	13.9	2015	1228	1750	14.6	0.91	+, Some surface pitting
	286	55	15.2	(2.2)	13.2	1915	1211	1720	13.9	0.87	-
	286	55	15.2	(2.2)	12.5	1815	1194	1690	13.5	0.84	-
Kalrez Compound 1045 (Dupont ECD-006)	287	57	101	14.7	11.1	1615	553	536	172.4	10.76	++, Specimen burned
	286	55	101	14.7	7.00	1015	519	475	115.3	7.20	+, Very slight surface change
	286	55	101	14.7	6.31	915	513	463	105.2	6.57	+, Very slight surface change
	287	57	101	14.7	5.62	815	504	448	95.5	5.96	-
	287	57	33.8	4.9	6.31	915	583	590	92.6	5.78	+, Slight surface change
	287	57	33.8	4.9	5.62	815	575	575	83.6	5.22	+, Very slight surface change
	287	57	33.8	4.9	4.93	715	565	558	74.6	4.66	+, Slight surface change
	287	57	33.8	4.9	4.24	615	553	535	65.7	4.10	-
	287	57	33.8	4.9	2.86	415	529	492	46.3	2.89	-
	287	57	33.8	4.9	1.48	215	486	416	26.1	1.63	-
	287	57	15.2	(2.2)	13.9	2015	1233	1760	14.4	0.90	+, Slight surface change

TABLE 2.5-2 (cont.)

Material	Initial Condition				Final Condition						Test Results
	Temperature		Pressure		Pressure		Calculated Temperature		Calculated Density		
	°K	°F	kN/m2	psia	MN/m2	psia	K	F	kg/m3	lb/ft3	
Kalrez Compound 1045 (Dupont ECD-006) (cont.)	288	58	15.2	(2.2)	11.8	1715	1183	1670	12.8	0.80	+, Very slight surface change
	289	60	15.2	(2.2)	11.1	1615	1166	1640	12.3	0.77	-
Epoxy EA934	293	67	101	14.7	5.62	815	514	465	93.5	5.84	+, Slight surface change
	286	55	101	14.7	4.24	615	486	415	74.6	4.66	+, Slight surface change
	286	55	101	14.7	3.55	515	475	395	63.9	3.99	+, Very slight surface change
	286	55	101	14.7	2.86	415	461	370	53.2	3.32	-
	286	55	33.8	4.9	1.48	215	484	412	26.3	1.64	+, Slight surface change
	286	55	33.8	4.9	0.79	115	447	345	15.2	0.95	+, Slight surface change
	286	55	33.8	4.9	0.45	65	415	287	9.3	0.58	-
	297	75	15.2	(2.2)	13.9	2015	1272	1830	14.1	0.88	+, Color change, surface melt
	286	55	15.2	(2.2)	7.00	1015	1016	1370	8.8	0.55	+, Very slight surface change
	286	55	15.2	(2.2)	6.31	915	983	1310	8.2	0.51	-

TABLE 2.5-3

OBSERVATIONS OF A NICKEL-200 SAMPLE WHICH WAS REPEATEDLY SUBJECTED TO ADIABATIC COMPRESSION OF GASEOUS NITROGEN TRIFLUORIDE

Test No.	Initial Pressure kg/m^2	Initial Pressure psia	Final Pressure MN/m^2	Final Pressure psia	Final Temperature K	Final Temperature F	
1	33.8	4.9	9.07	1315	611	641	+, Some surface change
2	33.8	4.9	9.07	1315	611	641	+, Slight additional change
3	33.8	4.9	9.07	1315	611	641	+, Slight additional change
4	33.8	4.9	9.07	1315	611	641	+, More change
5	33.8	4.9	9.07	1315	611	641	+, Some pitting
6	33.8	4.9	9.07	1315	611	641	+, More Change
7	33.8	4.9	9.07	1315	612	643	+, More change
8	33.8	4.9	9.07	1315	612	643	+, More change
9	33.8	4.9	9.07	1315	612	643	+, More change
10	33.8	4.9	9.07	1315	612	643	+, More change
11	33.8	4.9	13.9	2015	644	700	+, Pitting
12	33.8	4.9	13.9	2015	644	700	+, Additional pitting

TABLE 2.5-4

SUMMARY OF UPPER-LIMIT VALUES FOR NO REACTIVITY BETWEEN VARIOUS MATERIALS AND GASEOUS NITROGEN TRIFLUORIDE DURING ADIABATIC COMPRESSION

INITIAL CONDITIONS:	NF_3, 101 kN/m^2 (14.7 psia) Ambient Temperature				NF_3, 33.8 kN/m^2 (4.9 psia) Ambient Temperature				15% NF_3/Ar, 101 kN/m^2 (14.7 psia) Ambient Temperature			
FINAL CONDITIONS:	Temp		Pressure		Temp		Pressure		Temp		Pressure	
Material	K	F	MN/m^2	psia	K	F	MN/m^2	psia	K	F	MN/m^2	psia
Aluminum 2219, T-87	551	532	9.76	1415	517	470	2.17	315	1183	1670	11.1	1615
Stainless Steel 304L*	576	577	13.9	2015	649	708	13.9	2015	1255	1800	13.9	2015
Stainless Steel 347	570	567	13.2	1915	560	548	4.24	615	1200	1700	12.5	1815
Stainless Steel 17-4PH	557	542	10.4	1515	469	385	1.14	165	1205	1710	11.8	1715
Inconel 718	514	465	5.62	815	536	506	2.86	415	1244	1780	13.2	1915
Monel 400	524	483	7.00	1015	490	423	1.48	215	1255	1800	13.9	2015
Nickel 200	572	570	13.2	1915	610	639	8.38	1215	1272	1830	13.9	2015
Titanium 6Al-4V	460	368	2.86	415	415	287	0.45	65	1005	1350	6.31	915
1010 Steel	513	463	5.62	815	457	363	0.79	115	1033	1400	7.00	1015
Copper OFHC	522	480	7.00	1015	<420	<297	<0.45	<65	1105	1530	8.38	1215
Polytetrafluoroethylene	510	458	4.93	715	542	515	2.86	415	1102	1525	8.38	1215
Kel-F 81 CTFE	521	478	7.00	1015	508	455	2.17	315	1144	1600	9.76	1515
Carbon CJPS	562	552	11.1	1615	562	552	4.24	615	1211	1720	13.2	1915
Kalrez Compound 1045 (Dupont ECD-006)	504	448	5.62	815	553	535	4.24	615	1166	1640	11.1	1615
Epoxy (EA-934)	461	370	2.86	415	415	287	0.45	65	983	1310	6.31	915

*No positive reactions were observed with the 304L.

2.5, Adiabatic Compression Tests (cont.)

The data also provide an insight into the conditions of compression which produce the greatest interaction with materials. The two significant items are as follows: (1) the reaction thresholds as a function of driving pressure decrease as the initial pressure of the nitrogen trifluoride decreases, and (2) dilution of the nitrogen trifluoride with an inert gas, in almost every case, increases the driving pressure threshold even though the calculated final temperatures due to adiabatic compression may be substantially increased by the dilution.

2.6 MECHANICAL IMPACT TESTS

The objective of the mechanical impact tests was to determine the effect of mechanical impact on materials in the presence of liquid and gaseous nitrogen trifluoride. Historically the test method was developed for evaluation of the compatibility of liquid oxygen with various materials. Experience demonstrated that materials which could withstand a mechanical impact at an energy level of 10 kg-m (72 ft-lbs) in liquid oxygen without reaction were suitable for use in liquid oxygen service.

2.6.1 Mechanical Impact in Liquid Nitrogen Trifluoride

The discussion is presented in two sections: (1) Apparatus and Procedures and (2) Experimental Results.

2.6.1.1 Apparatus and Procedures

The mechanical impact tests were conducted in accordance with ASTM D-2512-70 (Reference: Annual Book of ASTM Standards, Part 18, 1973). This method is normally used to determine the relative sensitivity of materials with liquid oxygen under impact energy using the Army Ballistic Missile Agency (ABMA) type tester. A sample of the test material is placed in an aluminum specimen cup and the 17-4 PH striker pin is centered in the cup which contains the liquid nitrogen trifluoride. The entire anvil assembly is cooled with liquid nitrogen to retain the liquid nitrogen trifluoride in the cup. The plummet is dropped from selected heights up to 1.22 meters (4 feet) onto the pin, which transmits the energy to the test specimen. The plummet weight is 9.07 $\pm$ .023 kg (20 $\pm$ 0.05 lb), and dropping it 1.22 meters (4 feet) provides the impact level of 11.07 kg-m (80 ft-lbs).

Observation for any reaction is made and the liquid nitrogen trifluoride impact sensitivity of the test material is noted. Drop tests are continued using fresh cups and striker pins for each drop until a threshold value is achieved or no sensitivity is noted up to 11 kg-m (80 ft-lbs) of impact energy. The approximate threshold value is defined as the greatest height at which no reaction is obtained in 20 drops.

A photograph of the test apparatus is shown in Figure 2.6.1 and a photograph of the anvil section of the apparatus for the testing with liquid nitrogen trifluoride is shown in Figure 2.6.2. The apparatus was enclosed in an acrylic plastic box which was continuously purged with nitrogen to prevent condensation of atmospheric vapors in the chilled portion of the apparatus. The drop times were measured with a special electronic timer which was triggered and stopped by electrical shorting contacts. Valid tests are those in which the actual plummet drop time corresponds to a value that is within 3 percent of the computed free-fall time.

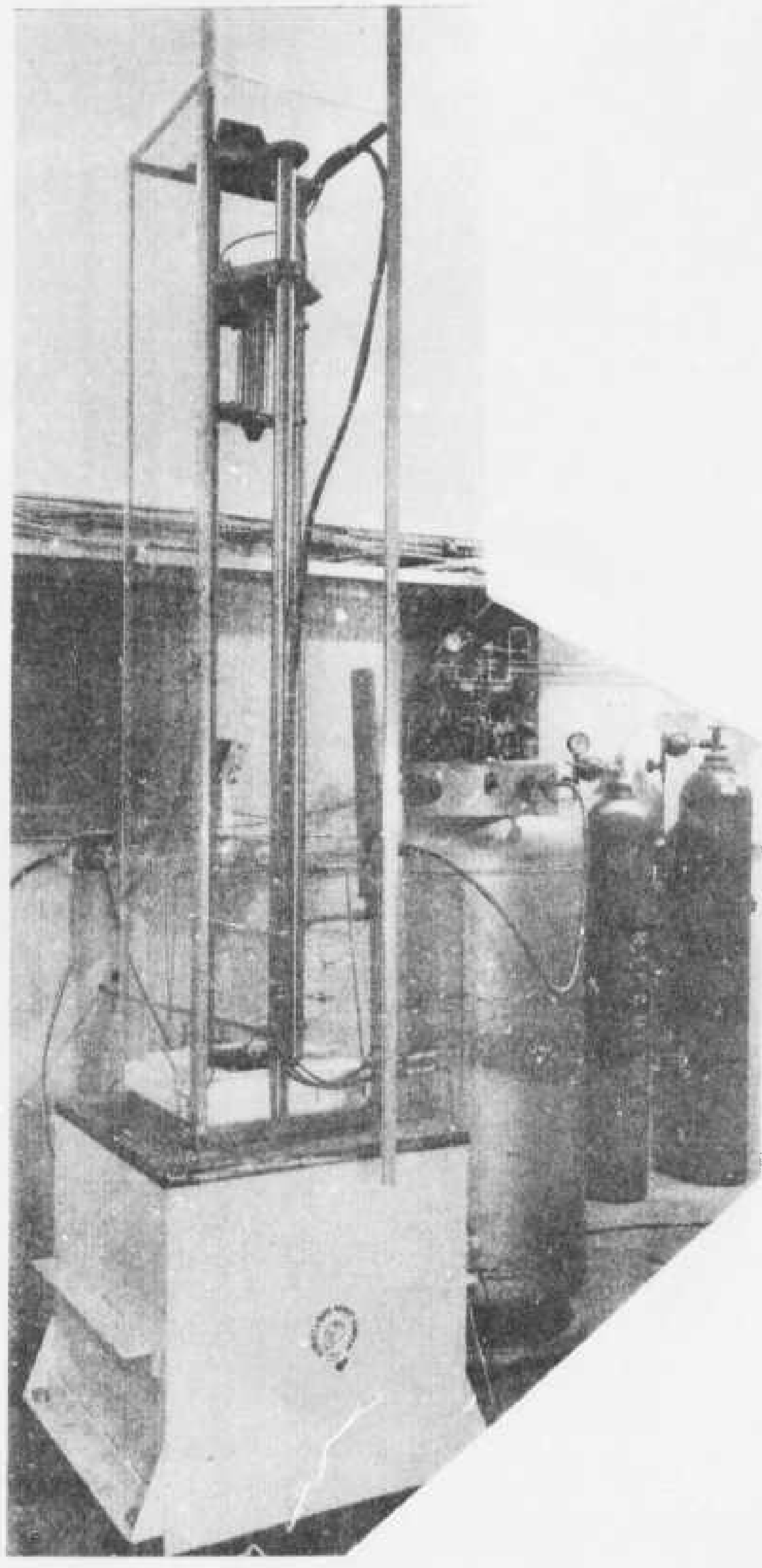


Figure 2.6.1. Photograph of the Mechanical Impact Tester

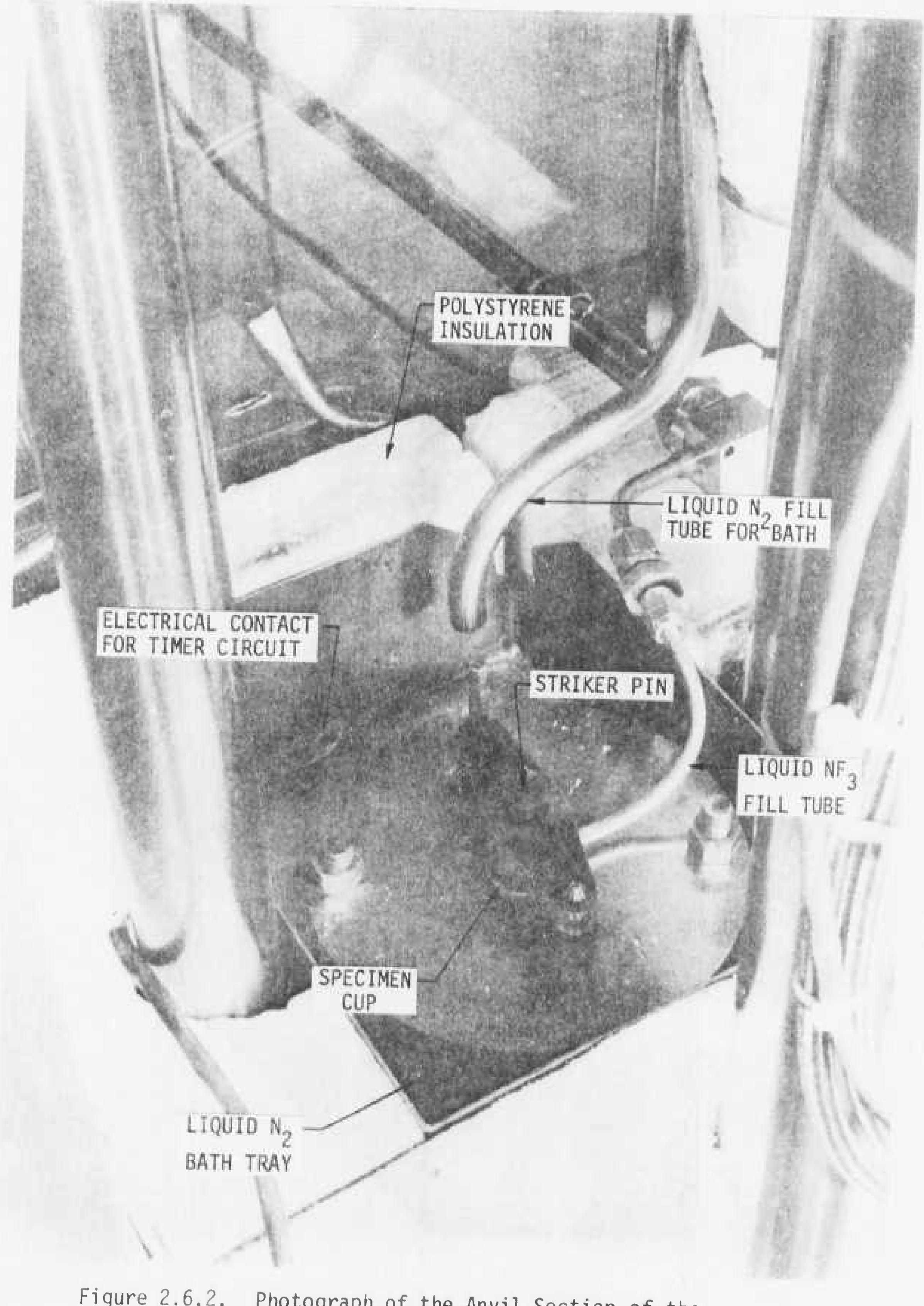


Figure 2.6.2. Photograph of the Anvil Section of the Mechanical Impact Tester

2.6, Mechanical Impact Tests (cont.)

2.6.1.2 Experimental Results

Based on the available literature for liquid fluorine, liquid chlorine pentafluoride, and liquid oxygen, only two types of metal alloys were appropriate candidates for impact testing, an aluminum alloy and a titanium alloy. The alloys selected for testing were 2219 aluminum, T-87, and 5Al-2.5 Sn titanium, ELI. The 5Al-2.5 Sn titanium was selected instead of 6Al-4V titanium because the former is more suitable for cryogenic service than the latter.

The non-metal candidates selected for testing were: polytetrafluoroethylene, Kel-F 81 CTFE, PFA teflon, and Viton. In addition a few tests were conducted with other non-metallic materials to obtain some indication of their reactivity in liquid nitrogen trifluoride under impact conditions. The data obtained from the tests are presented in Table 2.6-1.

The significant items to note from the data are as follows. The threshold energy-level values for reaction are greater than 11 kg-m (80 ft-lb) for the polytetrafluoroethylene and Kel-F 81 CTFE and this corresponds to the maximum energy level attainable with the apparatus. The threshold energy level for Viton, Class 1 is 6.9 kg-m (50 ft-lb). The threshold energy-level for the PFA Teflon is 9.7 kg-m (70 ft-lb). For comparison purposes, energy-level values for polytetrafluoroethylene and Kel-F 81 in liquid oxygen are greater than 10 kg-m (72 ft-lbs) (the upper limit tested). Viton A was found to vary from batch to batch in reactivity and 5 kg-m (36 ft-lbs) was an apparent threshold value. No data were found for PFA Teflon in liquid oxygen. For the 2219 aluminum, the threshold energy-level value in liquid nitrogen trifluoride was found to be greater than 11 kg-m (80 ft-lb); for the 5 Al-2.5 Sn titanium, the threshold energy-level value is 10 kg-m (72 ft-lb). In all the tests in liquid NF_3, the positives were noted as a flash of light.

For comparison purposes, the energy-level value for reaction between liquid oxygen and 2219 aluminum is greater than 10 kg-m (72 ft-lbs), and for 6Al-4V titanium the value is approximately 2 kg-m (14 ft-lbs).

The limited tests with the silicone grease indicate that its threshold energy-level value is less than 2 kg-m (15 ft-lb) and sustained burning was observed along with the flash of light. One test with Krytox at the 11 kg-m (80 ft-lb) energy level produced a negative result. Two tests were conducted with polyethylene at the maximum drop height, 1.22 meters (48 inches). One drop was negative, but the other produced a flash of light with sustained burning. One test was conducted with

TABLE 2.6-1

EFFECTS ON VARIOUS MATERIALS SUBJECTED TO MECHANICAL IMPACT IN LIQUID NITROGEN TRIFLUORIDE AT 77K

Material	Material Thickness mm	Material Thickness inch	Drop Height m	Drop Height inches	Results Positives	Results No. of Drops
Polytetrafluoroethylene	1.63	.064	1.22	48	0	20
Kel-F-81 CTFE	0.81	.032	1.22	38	0	20
Viton, Class 1 (Parco 9009-75)	2.2	.085	1.22	48	1	6
			1.10	43.3	1	2
			0.84	33	2	7
			0.76	30	0	20
			0.61	24	0	20
PFA Teflon	.18	.007	1.22	48	1	14
			1.14	45	1	5
			1.10	43.3	1	4
			1.07	42	0	20
			0.99	39	0	20
			0.84	33	0	20
2219 Aluminum, T-87	1.37	.054	1.22	48	0	20
5Al-2.5 Sn Titanium, ELI	0.81	.032	1.22	48	1	11
			1.14	45	1	12
			1.10	43.3	0	21
			.84	33	0	3
Silicone Grease (Dow Corning High Vacuum)			.30	12	2	2
			.23	9	1	1
			.15	6	0	1

Vaseline at the maximum drop height; the result was a detonation which shattered the plexiglas box which surrounded the apparatus. These tests re-emphasized the necessity for maintaining the NF_3 systems free of hydrocarbon contamination.

The composition of the nitrogen trifluoride used in the tests was as follows:

Component	Content, Weight Percent
NF_3	99.66
Active fluorides as HF	0.0006
N_2	0
CO/O_2	0.29
CF_4	0.017
CO_2	0.011
N_2O	0.017

2.6.2 Mechanical Impact in Gaseous Nitrogen Trifluoride

Because high pressure storage of nitrogen trifluoride is one means to minimize the storage container size, mechanical impact tests were conducted with potential non-metallic materials which would be used in such systems at pressures up to 17.34 MN/m^2 (2500 psig). The discussion is presented in two sections: (1) Apparatus and Procedures, and (2) Experimental Results.

2.6.2.1 Apparatus and Procedures

The tests were conducted in a manner similar to those involving gaseous oxygen which have been described by C. F. Key (Reference 2.6.1). The impact test apparatus is shown in Figure 2.6.3. Because the tests were conducted at ambient temperatures, the acrylic plastic box which enclosed the test apparatus was not required to prevent moisture condensation and therefore was removed to provide better access to the test components.

The anvil section which contained the nitrogen trifluoride at high pressures is shown in a photograph in Figure 2.6.4. The schematic of the anvil design is presented in Figure 2.6.5. High pressure nitrogen was used to counterbalance the effect of the nitrogen trifluoride pressure against the striker pin. Teflon O-rings were used to

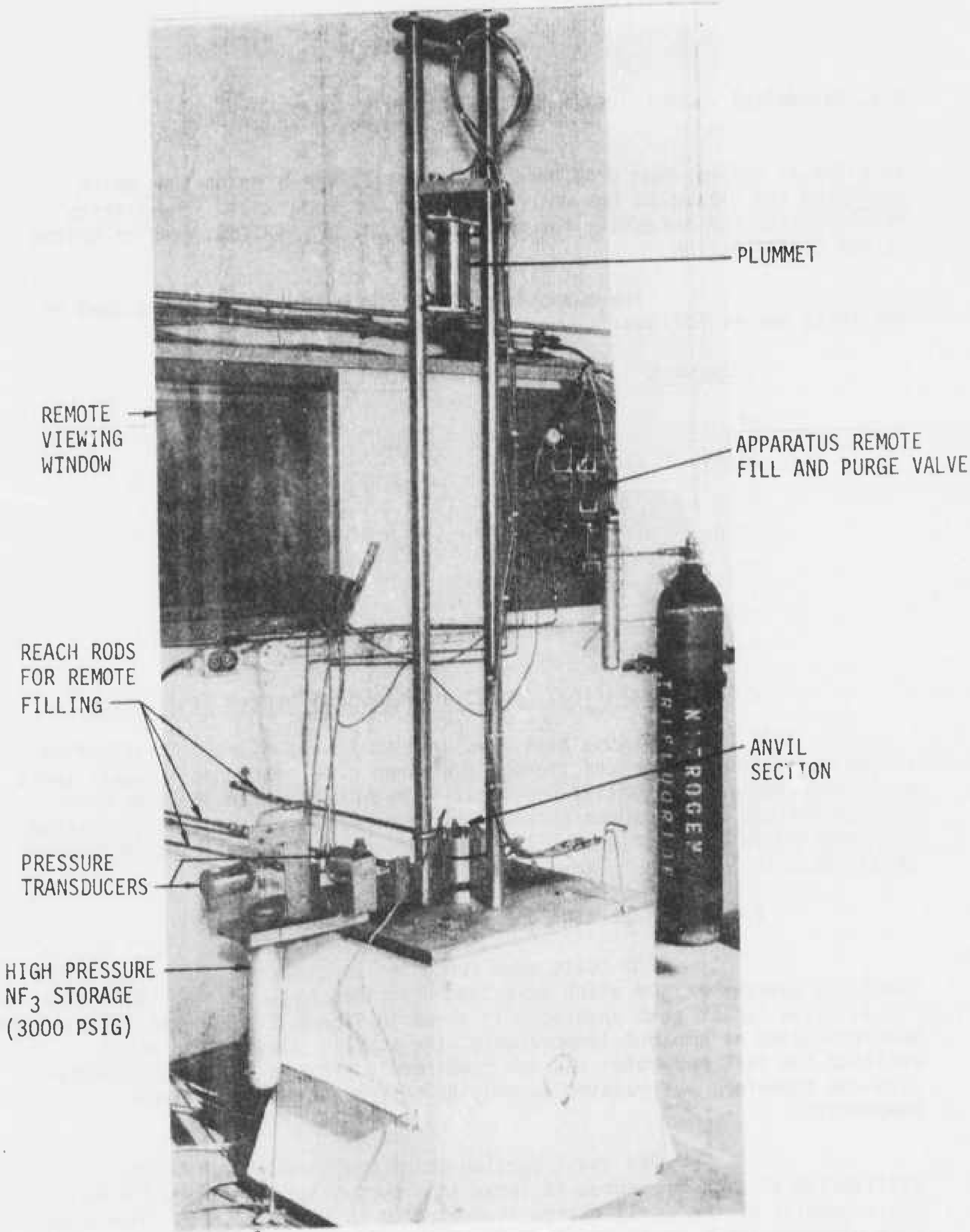


Figure 2.6.3. Mechanical Impact Test Apparatus for Gaseous Nitrogen Trifluoride Environment

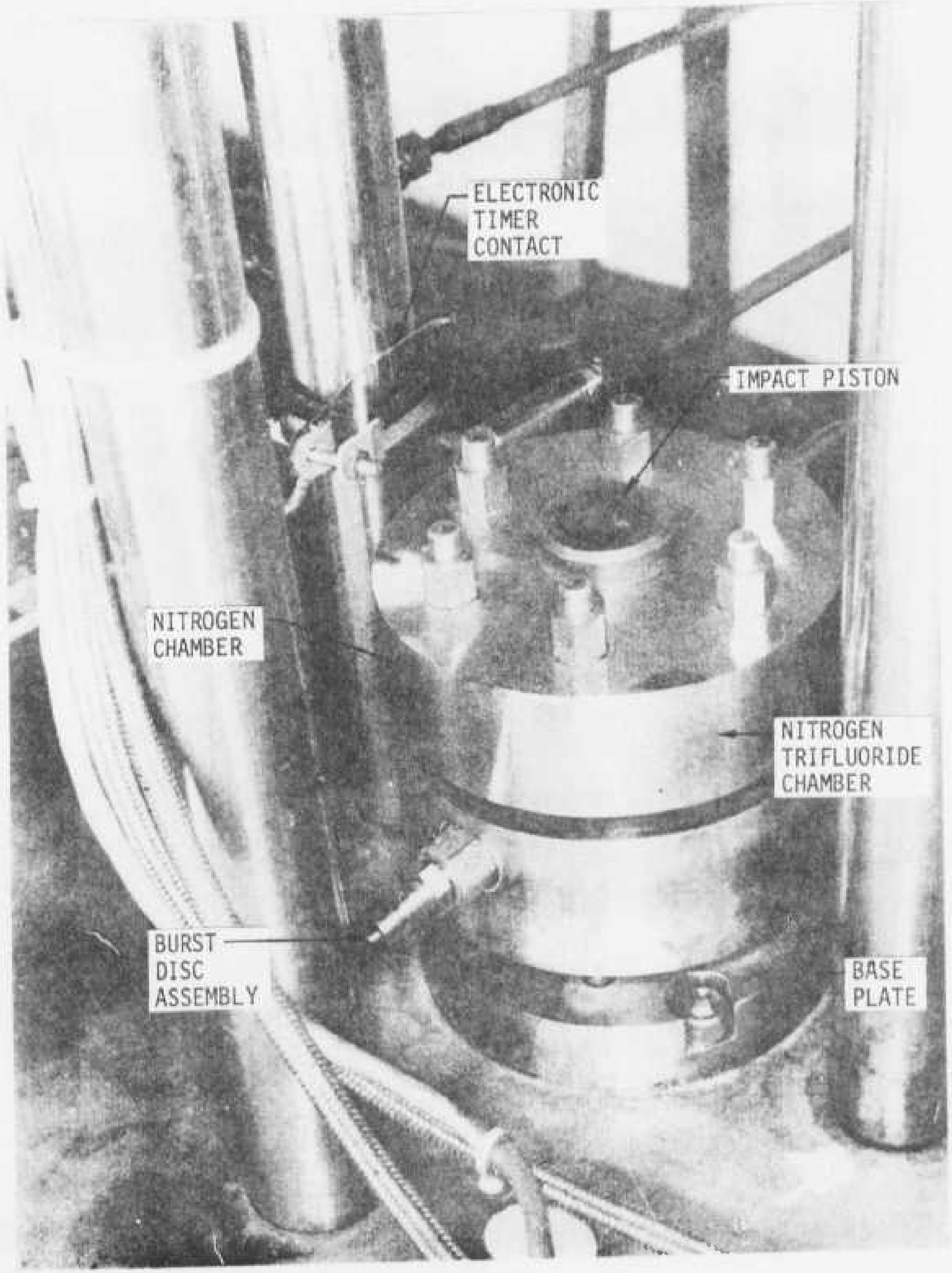


Figure 2.6.4. Anvil Section for High Pressure Gas Testing with the Mechanical Impact Tester

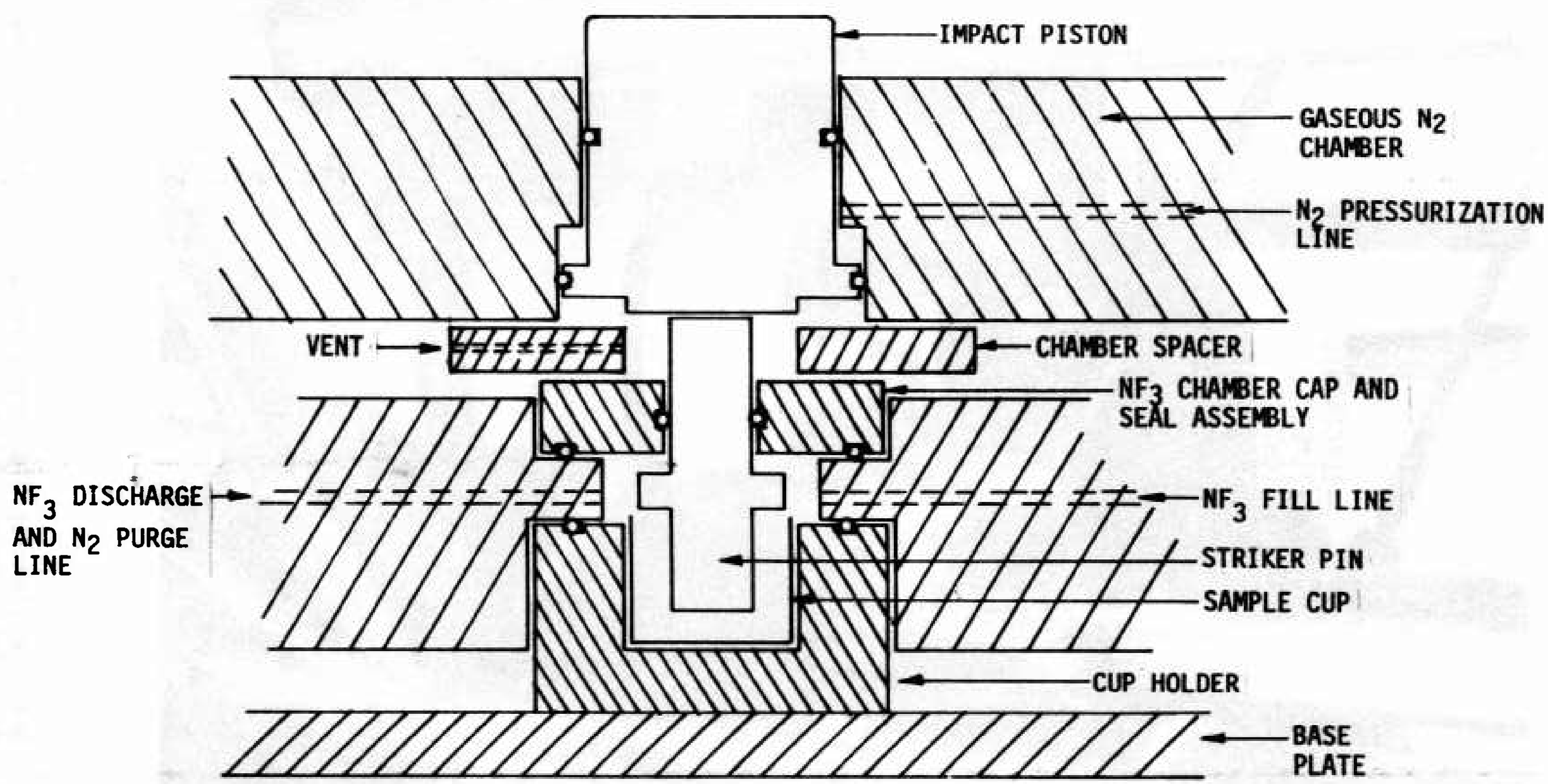


Figure 2.6-5. Schematic Diagram of High Pressure Gaseous Nitrogen Trifluoride Impact Tester Anvil

seal the gas chambers. A rupture disc was incorporated in the NF_3 chamber to prevent the pressure from exceeding 24.23 MN/m^2 (3500 psig). The threshold levels for reaction were identified in the same manner as described for the mechanical impact tests in liquid nitrogen trifluoride. The positive reactions all resulted in test samples being consumed and usually the burst disc ruptured.

2.6.2.2 Experimental Results

Only non-metallic materials were tested in gaseous nitrogen trifluoride. The materials were polytetrafluoroethylene, Kel-F 81 CTFE, PFA Teflon, and Viton, Class 1 (Parco 9009-75). The materials were tested at pressure levels as great as 17.34 MN/m^2 (2500 psig) and as low as 7.0 MN/m^2 (1000 psig). The data are presented in Table 2.6-2.

Based on the data, the threshold energy-level values for reaction are (1) 3.45 kg-m (25 ft-lbs) at 7.0 MN/m^2 (1000 psig) for PFA Teflon, (2) 2.72 kg-m (20 ft-lbs) at 7.0 MN/m^2 (1000 psig) for Kel-F 81, (3) greater than 11 kg-m (80 ft-lbs) at 7.0 MN/m^2 (1000 psig), 9.0 kg-m (65 ft-lbs) at 8.72 MN/m^2 (1250 psig), about 3.45 kg-m (25 ft-lbs) at 17.34 MN/m^2 (2500 psig) for polytetrafluoroethylene, and (4) greater than 11 kg-m (80 ft-lbs) at 17.34 MN/m^2 (2500 psig) for the Viton, Class 1. The latter result is surprising in view of Viton's behavior in liquid NF_3 and may be due to the ability of the elastomer to dissipate the impact energy at ambient temperatures.

Two different cylinders of nitrogen trifluoride were used in the tests. The compositions were as follows.

	Composition, Weight Percent						
	NF_3	Active Fluorides as HF	N_2	CO/O_2	CF_4	CO_2	N_2O
Cylinder H79957	99.01	<.0001	0	0.27	0.65	.012	.053
Cylinder 309029	99.58	<.0001	.039	0.30	0.030	.0083	.043

As a basis for comparison with oxygen, Key has reported that in gaseous oxygen at 6.8 MN/m^2 (986 psia) samples of polytetrafluoroethylene 0.157 cm thick reacted in 20 percent of the impact drops at an energy level of 10 kg-m (72 ft-lbs) and samples 0.086 cm thick reacted in 10 percent of the impact drops at an energy level of 10 kg-m (72 ft-lbs) (Reference 2.6.1).

TABLE 2.6-2

EFFECTS ON NON-METALLIC MATERIALS SUBJECTED TO MECHANICAL IMPACT IN GASEOUS NITROGEN TRIFLUORIDE AT AMBIENT TEMPERATURES

Materials	Material Thickness		Gaseous NF_3 Pressure		Drop Height		Results	No. of
	mm	Inch	MN/m^2	psia	m	Inches	Positives	Drops
Polytetrafluoroethylene	1.63	.064	17.34	2515	1.22	48	1	4
			17.34	2515	1.10	43.3	1	1
			17.34	2515	0.84	33	1	1
			17.34	2515	0.61	24	1	1
			17.34	2515	0.38	15	0	8
			10.45	1515	1.22	48	1	2
			8.72	1265	1.22	48	1	2
			8.72	1265	0.84	43.3	1	1
			8.72	1265	1.07	42	0	1
			8.72	1265	0.99	39	0	20
			8.72	1265	0.84	33	0	20
			7.0	1015	1.22	48	0	20
Kel-F 81 CTFE	0.81	.032	17.34	2515	0.84	33	1	1
			17.34	2515	0.61	24	1	3
			17.34	2515	0.38	15	0	2
			7.00	1015	0.84	33	1	1
			7.00	1015	0.61	24	1	2
			7.00	1015	0.38	15	1	1
			7.00	1015	0.30	12	0	20
			7.00	1015	0.15	6	0	20
PFA Teflon	0.18	.007	7.00	1015	1.10	43.3	1	1
			7.00	1015	0.84	33	1	1
			7.00	1015	0.61	24	1	6
			7.00	1015	0.53	21	1	6
			7.00	1015	0.46	18	1	1
			7.00	1015	0.38	15	0	20
Viton, Class 1 (Parco 9009-75)	2.2	.085	17.34	2515	1.22	48	0	20
			13.89	2015	1.22	48	0	1
			10.45	1515	1.22	48	0	1
			7.00	1015	1.22	48	0	1
			7.00	1015	1.10	43.3	0	1
			7.00	1015	0.61	24	0	2
			7.00	1015	0.38	15	0	1
			7.00	1015	0.30	12	0	1
			7.00	1015	0.15	6	0	1

2.7 FLOW IMPACT TESTS

The objective of the liquid impact tests was to determine the maximum temperature to which various materials may be heated without detrimental effects occurring when impacted by liquid streams of nitrogen trifluoride. The tests were performed at two velocities to establish the effect of velocity on the allowable temperature level. The materials tested were

Al 2219, T-87	Ti 6Al-4V, STA
CRES 304L, Annealed	1010 Steel, Normalized
CRES 347, Annealed	Cu OFHC, Annealed
CRES 17-4 PH, H-1025	Polytetrafluoroethylene
Inconel 718, STA	Kel-F 81 CTFE
Monel 400, Annealed	Carbon CJPS
Nickel 200, Annealed	PFA Teflon

The apparatus, procedures, and results are discussed below.

a. Apparatus and Procedures

The test apparatus consisted of two major components: (1) a liquid feed system which was maintained at 77 K (-196 C) and (2) an electrical-resistance heating system for the metal specimens. A photograph of the test apparatus is shown in Figure 2.7.1. The temperature of the metal specimens was measured by means of thermocouples attached to the back of the metal strip which was approximately .25 mm (0.010-in.) thick (Figure 2.7.2). The metal specimens were placed within 4.76 mm (3/16 in.) of the discharge orifice of the liquid propellant feed system. The exit orifice diameter of the propellant feed system was approximately 0.38 mm (0.015 in.) and the entire feed system was appropriately temperature conditioned with liquid nitrogen. For each test, a fresh, unpassivated specimen was used and the temperature level was increased in 56 K (100 F) increments for each test until the impacting liquid propellant caused the metal specimen to burn. As soon as the metal specimen reached the desired temperature, the liquid propellant was flowed for one second during each test and the feed lines were then immediately purged with helium to prevent moisture from accumulating at the exit orifice. The temperature at which significant attack occurs was determined within approximately 33 K (50 F). Normally there was only slight evidence of attack on the metal surfaces until the ignition temperature was reached.

Nonmetallic materials were placed on a metal strip which was heated electrically. A non-metal specimen of PFA Teflon is shown in Figure 2.7.3. The non-metal specimen was held in place by means of two

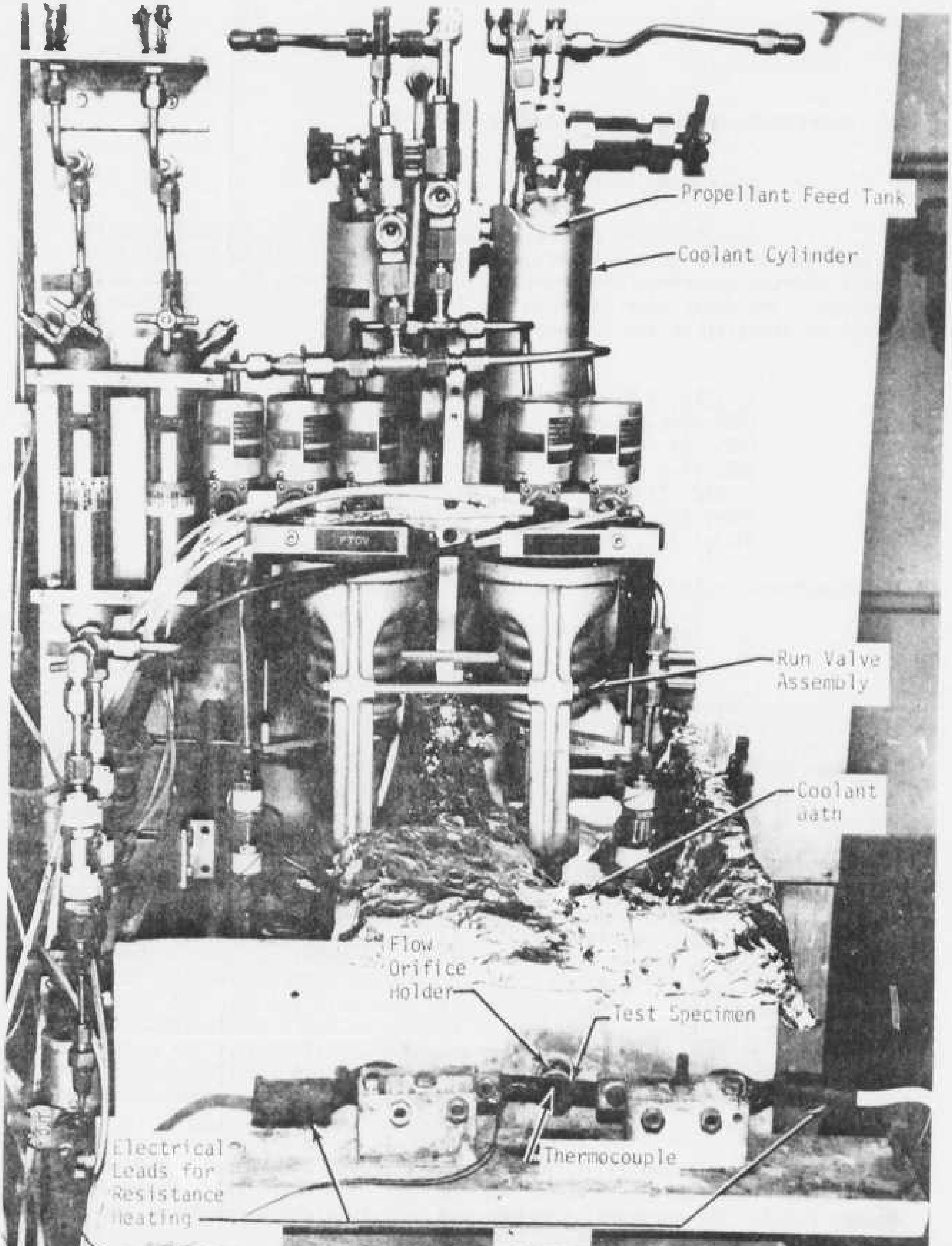


Figure 2.7.1. Apparatus for Flow Impact Tests

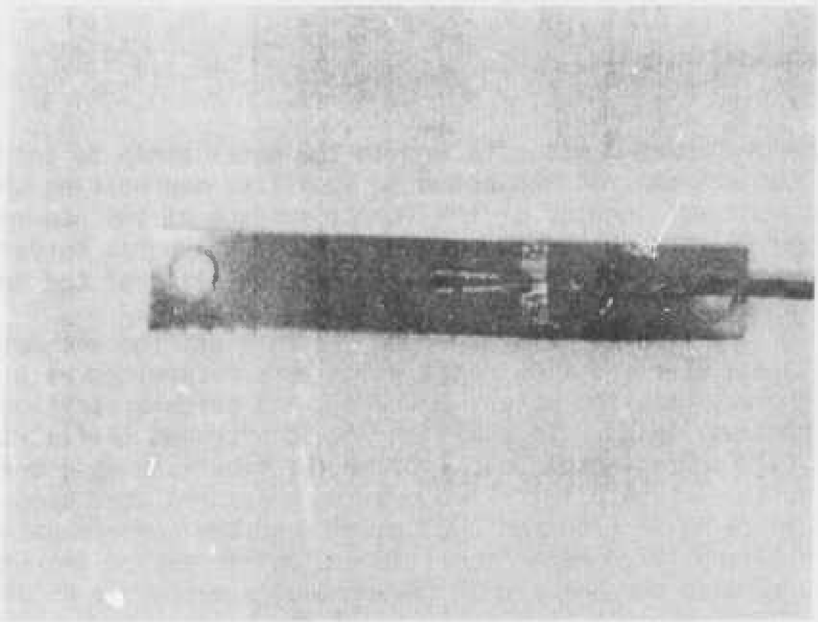


Figure 2.7.2. Metal Specimen for Liquid Impact Testing with Thermocouple Attached to Back Surface

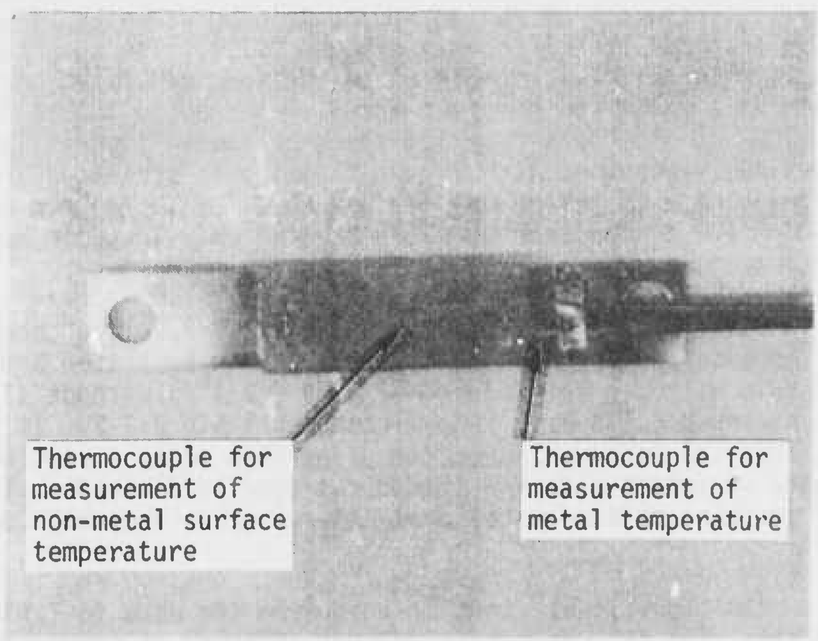


Figure 2.7.3. Non-Metal Specimen for Liquid Impact Testing with Thermocouples Attached

wire loops. One thermocouple was attached to the metal strip to insure that the non-metal specimen was not subjected to localized overheating while another thermocouple was located at the impact surface of the non-metal specimen in order to measure the temperature of the non-metal surface. The latter thermocouple values were used in the tabulation of the data.

The flow velocity through the exit orifice was determined from the orifice diameter and flow rates which were determined as a function of driving pressure using water, methanol, and trichlorotrifluoroethane as calibration fluids. In addition liquid nitrogen trifluoride itself at 77 K (-196 C) was used to calibrate the flow rate at a driving pressure of 1.82 MN/m^2 (250 psig). Two driving pressures were used in the tests 1.82 and 13.79 MN/m^2 (250 and 2000 psig), and the corresponding velocities were 39 and 107.6 meter/sec (128 and 353 ft/sec). The temperatures were measured with chromel-alumel thermocouples which are accurate to $\pm$ 0.75% or approximately $\pm$ 11 K ($\pm$ 20 F) at the highest temperatures encountered.

b. Experimental Results

The results of the liquid nitrogen trifluoride impacting the heated metal and non-metal specimens are reported as either being positive or negative test results. A photograph containing examples of positive and negative test results is shown in Figure 2.7.4. The span between the temperature at which significant attack of the specimen first occurred and the temperature at which the specimen actually burned on impact is less than 56 K (100 F). The data from 136 tests are summarized in Table 2.7-1. Data previously obtained with liquid fluorine is presented in the table for the convenience of the reader (Reference 2.7.1). The non-metal specimens except for the carbon specimen were treated only to their normal maximum usage temperature.

Based on the data summarized in Table 2.7-1, a tabulation of the maximum temperatures to which metal surfaces can be heated and then impacted with liquid nitrogen trifluoride at 77°K (-321°F) without significant attack was prepared. The data is presented in Table 2.7-2. It should be kept in mind that the data was generated using thin metal strips which are readily cooled on impact with the liquid nitrogen trifluoride. The threshold level for more massive metal parts may occur at somewhat lower temperatures.

The significant items to note from the data in Tables 2.7-1 and 2.7-2 are: (1) the non-metals are not attacked at temperatures corresponding to their maximum usage temperature by the impacting liquid nitrogen trifluoride, (2) the reaction threshold between liquid nitrogen

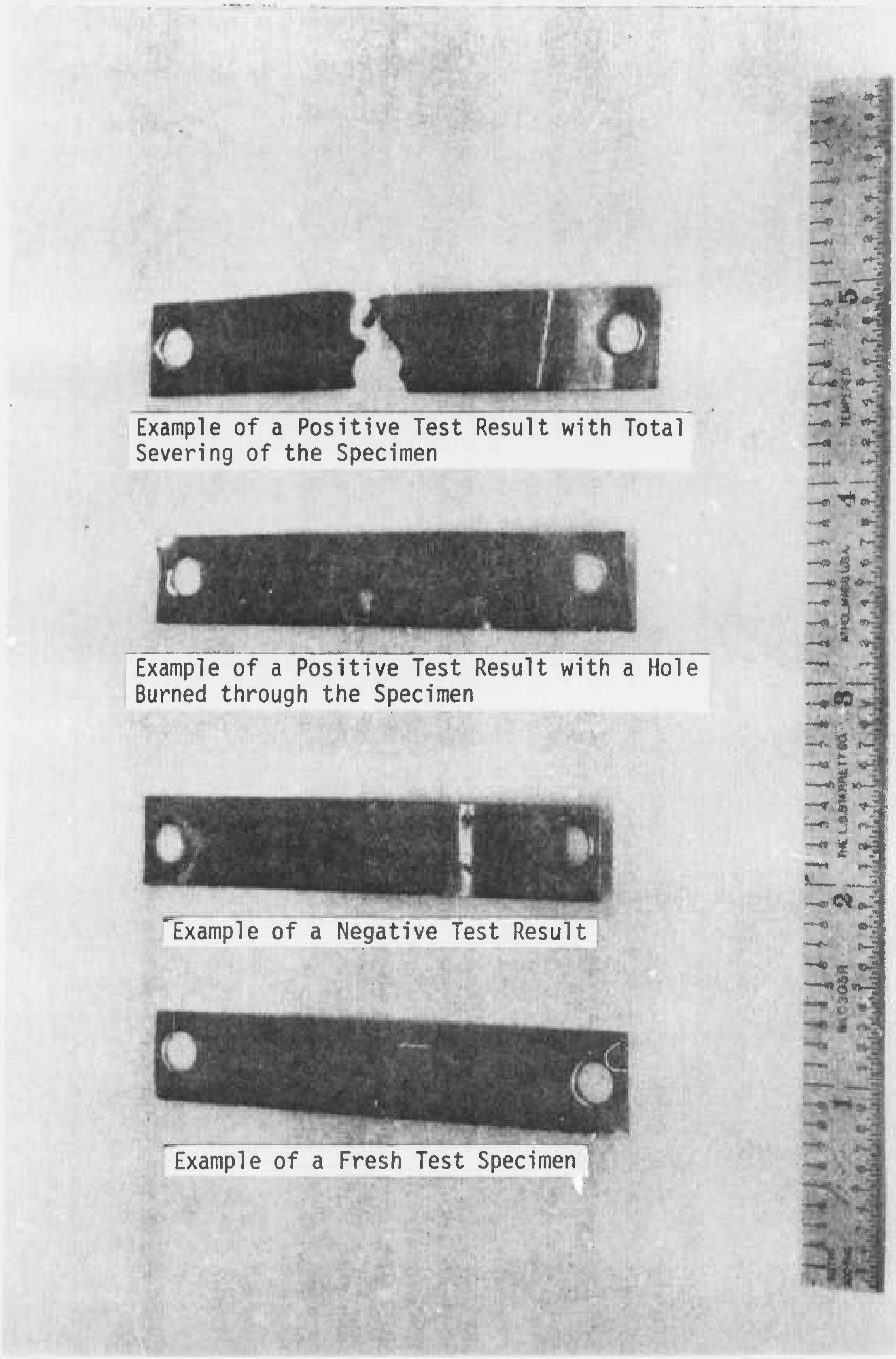


Figure 2.7.4. Typical Metal Specimens from Liquid Impact Tests

TABLE 2.7-1

DATA INDICATIVE OF THE REACTIVITY OF LIQUID NITROGEN TRIFLUORIDE AND LIQUID FLUORINE AT 77°K IMPACTING ON VARIOUS HEATED MATERIALS

Specimen Material	Initial Specimen Temperature When Impacted by Liquid Nitrogen Trifluoride — Liquid Velocity 107.6 m/s (353 ft/s)				Liquid Velocity 39 m/s (128 ft/s)				Initial Specimen Temperature When Impacted by Liquid Fluorine — Liquid Velocity 73 m/s (240 ft/s)		Liquid Velocity 39.6 m/s (130 ft/s)	
	Positive Reaction		No Reaction		Positive Reaction		No Reaction		Postive Reaction	No Reaction	Positive Reaction	No Reaction
	K	F	K	F	K	F	K	F	K	K	K	K
Aluminum 2219	Burned in air	> 900	755	≤ 900 (3)	Burned in air	∿ 900	755	≤ 900 (3)	839 (1)			728 (1)
CRES 304L	1366	2000 (1)	1339	≤1950 (3)	1339	≥1950 (2)	1311	≤1900 (2)	1200 (2)	1172 (2)	1200 (5)	1172 (4)
CRES 316L					1478	≥2200 (1)	1450	≤2150 (1)				
CRES 347	1255	≥1800 (4)	1228	≤1750 (2)	1255	≥1800 (3)	1228	≤1750 (5)	978 (1)	922 (2)	1033 (2)	978 (2)
CRES 17-4 PH	1366	≥2000 (4)	1339	≤1950 (1)	1478	≥2200 (2)	1450	≤2150 (3)				
Inconel 718	1394	≥2050 (2)	1366	≤2000 (4)	1339	≤1950 (2)	1311	≤1900 (2)	1339 (2)	1283 (1)	1366 (2)	1339 (2)
Monel 400	Burned in air	2250	1478	≤2200 (4)	1478	2200 (1)	1450	≤2150 (3)				
Nickel 200	1589	2400 (1)	1561	2350 (1)	1616	≥2450 (1)	1589	≤2400 (4)	1450 (1)	1422 (2)	1478 (3)	1450 (3)
Titanium 6Al-4V	1450	2150 (1)	1422	2100 (1)	1422	2100 (1)	1394	≤2050 (3)				
1010 Steel	1339	≥1950 (5)	1311	1900 (1)	1450	≥2150 (3)	1422	≤2100 (4)				
Copper OFHC	Burned in air	>1700	1200	1700 (1)	Burned in air	∿1700	1200	≤1700 (6)				
Polytetrafluoroethylene			533	≤ 500 (2)			533	≤ 500 (2)				
Kel F 81 CTFE			478	≤ 400 (2)			478	≤ 400 (2)				
Carbon CJPS			1255	≤1800 (1)			1255	≤1800 (2)				
PFA Teflon			478	≤ 400 (2)			478	≤ 400 (2)				

( ) indicates the number of specimens included in the data point.

TABLE 2.7-2

MAXIMUM TEMPERATURES OF METAL SURFACES ON WHICH IMPACTING STREAMS OF LIQUID NITROGEN TRIFLUORIDE AT 77 K DO NOT RESULT IN IGNITION

	Approximate Non-Ignition Threshold Temperature			
	Liquid Velocity 107.6 m/s (353 ft/s)		Liquid Velocity 39 m/s (128 ft/sec)	
Material	K	F	K	F
Aluminum 2219	755	900	755	900
CRES 304 L	1339	1950	1311	1900
CRES 316 L		--	1450	2150
CRES 347	1228	1750	1228	1750
CRES 17-4 PH	1339	1950	1450	2150
Inconel 718	1366	2000	1311	1900
Monel 400	1478	2100	1450	2150
Nickel 200	1561	2350	1589	2400
Titanium 6Al-4V	1422	2100	1394	2050
1010 Steel	1311	1900	1422	2100
Copper OFHC	1200	1700	1200	1700
Polytetrafluoroethylene	533	500	533	500
Kel F 81 CTFE	478	400	478	400
Carbon CJPS	1255	1800	1255	1800
PFA Teflon	478	400	478	400

trifluoride and the heated metals is higher than that between liquid fluorine and the heated metals and (3) the effect of velocity on the threshold value is minimal except for the 1010 steel and CRES 17-4 PH, both of which dropped 111°K (200°F) as the velocity increased from 39 and 107.6 meters/sec (128 to 353 ft/sec).

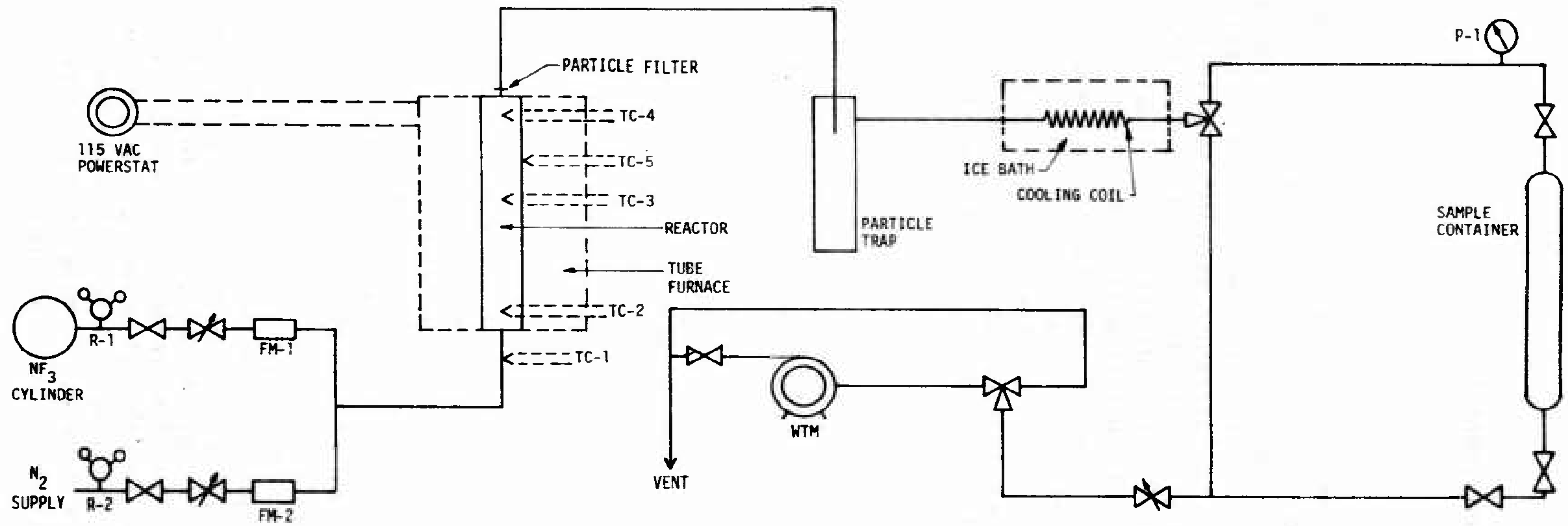
2.8 WASTE DISPOSAL TESTS

The objective of the waste disposal test series was to investigate methods for disposing of nitrogen trifluoride in an environmentally acceptable manner. Tests in the series were conducted by passing nitrogen trifluoride through packed static beds using both limestone and activated charcoal as candidate bed materials. No literature data was available concerning the reaction of limestone with nitrogen trifluoride, but previous investigations by Massonne (Reference 2.8.1) indicated that activated charcoal heated in the presence of nitrogen trifluoride could react explosively. Gould (Reference 2.8.2), however, reported no such problem when passing nitrogen trifluoride through a preheated fluidized charcoal bed. In our investigations, care was taken to preheat charcoal beds so that significant adsorption of nitrogen trifluoride by the bed would not occur and lead to the accumulation of the oxidizer prior to reaction initiation.

2.8.1 Apparatus and Procedure

A schematic diagram of the waste disposal apparatus is shown in Figure 2.8.1. Two different reactor types were employed during the tests. Both were externally heated one inch diameter tubes made of 304 stainless steel. The original reactor used in Tests 1 through 3 and 14 through 18 was 61 cm (24 in.) long with bed thermocouples at 7.6 cm (3 in.), 30.5 cm (12 in.), and 53.3 cm (21 in.) from the gas inlet. In order to detect hot spots in the reaction bed, a second reactor, 40.6 cm (16 in.) long, with bed thermocouples at 1.9 cm (.75 in.), 4.4 cm (1.75 in.), 5.7 cm (2.25 in.), and 20.3 cm (8 in.) from the gas inlet was used in runs 20 and 21.

At the start of each test, the bed material with nitrogen gas flowing through it was heated to a temperature sufficient for reaction initiation, 616 K (650 F) for limestone and 533 K (500 F) for activated charcoal. When this bed temperature was attained, nitrogen trifluoride was introduced into the system at a predetermined flow rate, while the system pressure was maintained at 0.14 MN/m^2 (5 psig) by adjusting the gas exit control valve. Bed temperatures were monitored during each test and temperature control was achieved through variation in nitrogen diluent flow. As bed temperatures began to stabilize, inlet gas driving pressures and system pressures were checked to insure that they remained at 1.4 MN/m^2 (50 psig) and 0.14 MN/m^2 (5 psig) respectively; nitrogen trifluoride and nitrogen flows were recorded from rotameters; reactor exit gas flow measurements were taken using a wet-test meter; and an effluent gas sample was trapped in the sample container. After obtaining the desired data, the nitrogen trifluoride flow was terminated and the reaction quenched by flooding the bed with nitrogen. Chemical analysis of the



FM-1	NF_3 ROTAMETER
FM-2	N_2 ROTAMETER
P-1	PRESSURE GAUGE, REACTOR EXIT
R-1	NF_3 REGULATOR
R-2	N_2 REGULATOR
TC-1	C-A THERMOCOUPLE, REACTOR INLET
TC-2	C-A THERMOCOUPLE, BED, 3" FROM REACTOR INLET
TC-3	C-A THERMOCOUPLE, BED, 12" FROM REACTOR INLET
TC-4	C-A THERMOCOUPLE, BED, 21" FROM REACTOR INLET
TC-5	C-A THERMOCOUPLE, REACTOR SKIN
WTM	WET TEST METER

Figure 2.8-1. Schematic Diagram of Waste Disposal Test Apparatus

effluent samples was performed by means of a Hewlett Packard 5830 A Gas Chromatograph in conjunction with active fluoride measurements from an Orion Model 96-09 fluoride specific ion electrode.

2.8.2 Experimental Results

Test results are presented in Table 2.8-1. In examining the data it should be carefully noted that temperatures given in the table represent the maximum measurement among thermocouple locations in the bed. Both limestone and activated charcoal static beds were found to react in extremely localized zones which were strongly influenced by the type of bed and gas velocity. This reaction behavior presented major difficulties in obtaining accurate reaction temperatures.

This reaction behavior also presented serious problems with the limestone bed itself. The temperature in the localized reaction zones was sufficient to cause sintering of the limestone particles which resulted in a consequent reduction of flow through the reactor. The problem was recognized by bed agglomerates deposited on the reactor wall during the short duration at conditions of Test 1. Attempts to control the temperature of the reaction zones through reduction in space velocity and an increase in diluent flow in Tests 2 and 3 respectively, resulted in continuing problems with the bed material.

Activated charcoal proved to be an advantageous choice for bed material in this application, not only due to its lack of agglomerative tendencies, but also because of its desirable nitrogen trifluoride conversion characteristics and its apparently safety when used in preheated beds.

No indication of any type of explosive tendency such as that reported by Massonne was observed when flowing nitrogen trifluoride through charcoal beds at the space velocities examined in this test series. Before introducing nitrogen trifluoride into the system, beds were heated to a minimum of 533 K (500 F) at which temperature reaction initiation was immediate.

Desirable conversion characteristics of activated charcoal beds include extremely low N_2F_4 production and excellent NF_3 conversion efficiency. Superior conversion efficiency is evident from the data in Table 2.8-1. None of the reaction conditions listed in the table for charcoal beds resulted in greater than 0.14% by volume unreacted nitrogen trifluoride in the exit gas. A direct comparison of nitrogen trifluoride conversion between limestone and charcoal beds under similar conditions is afforded by Tests 2 and 16. In both tests, undiluted

TABLE 2.8-1

WASTE DISPOSAL TEST DATA

Run	Inlet Gas Composition							Reaction Parameters					Exit Gas Composition							
Sample #	NF_3 Vol. %	N_2 Vol. %	CO/O_2 Vol. %	CF_4 Vol. %	CO_2 Vol. %	N_2O Vol. %	Active Fluoride Vol. %	Temp. (Max t.c. in bed) K	Pressure MN/m^2	Pressure psig	Velocity m/sec	Space Velocity sec^{-1}	NF_3 Vol. %	N_2 Vol. %	CO/O_2 Vol. %	CF_4 Vol. %	CO_2 Vol. %	N_2O Vol. %	Active Fluoride Vol. %	Bed Composition (Remarks)
1-1	56.44	43.10	0.39	0.0068	0	0.025	0.032	775	0.136	5	0.53	0.255	33.83	55.79	1.67	0.0063	6.43	0.093	2.18	$CaCO_3$ (Bed Agglomeration)*
2-1	99.20	0	0.69	0.012	0	0.044	0.056	727	0.136	5	0.063	0.056	32.62	1.91	18.64	0.014	28.11	0.44	18.26	$CaCO_3$ (Bed Agglomeration)
3-1	26.70	73.08	0.19	0.0032	0.012	0.010	0.0027	852	0.136	5	0.271	0.056	1.14	54.54	12.86	0.010	30.56	0.18	0.71	$CaCO_3$ (Bed Agglomeration)
14-1	14.37	85.35	0.099	0.076	0	0.0096	0.00087	1505	0.136	5	1.66	0.103	0.11	99.06	0	0.64	0.12	0.035	0.036	Activated Charcoal**
15-1	9.74	90.07	0.067	0.053	0	0.0065	0.00059	1078	0.136	5	1.76	0.103	0.066	98.68	0	1.17	0.058	0	0.023	Activated Charcoal
16-1	98.16	0.55	0.67	0.54	0	0.065	0.0060	1023	0.136	5	0.10	0.060	0.14	89.79	0	9.90	0.15	0	0.015	Activated Charcoal
17-1	9.38	90.45	0.064	0.052	0	0.0063	0.00057	1153	0.136	5	2.34	0.124	0.0020	97.59	0	2.34	0.049	0	0.017	Activated Charcoal
18-1	11.54	88.25	0.079	0.064	0	0.0077	0.00070	1329	0.136	5	2.20	0.124	$\le$0.0007	98.87	0	1.05	0.053	0	0.020	Activated Charcoal
20-1	11.54	88.25	0.079	0.064	0	0.0077	0.00070	1500	0.136	5	2.48	0.185	0.019	95.45	0	4.43	0.082	0	0.020	Activated Charcoal
21-1	11.95	87.82	0.082	0.065	0	0.0079	0.00074	>1366	0.136	5	>1.50	0.128	$\le$0.0007	97.36	0	2.59	0.032	0	0.016	Activated Charcoal

*The limestone ($CaCO_3$) was used in the form of 18-20 mesh particles.
**The activated charcoal was obtained from Culligan Water Conditioning Co., nominal 8 mesh was dried prior to use.

nitrogen trifluoride was fed into the reactor at a space velocity of about 200 hr^{-1}. The effluent gas stream from the limestone packed reactor in Test 2 showed 32.62% nitrogen trifluoride remaining, while the charcoal bed reactor stream in Test 16 contained only 0.14% by volume unreacted NF_3.

Optimum nitorgen trifluoride conversion was obtained by using between 11.5% and 12% nitrogen trifluoride inlet gas at a space velocity of about 450 hr^{-1}. At these conditions, no nitrogen trifluoride was detected in the effluent gas streams of Tests 18 and 20. An increase in space velocity from optimum conditions, as in Test 19, resulted in a very low content of unreacted nitrogen trifluoride in the exit gas. Decreasing the temperature in Tests 15 and 17 through increased dilution also resulted in very small but detectable amounts of nitrogen trifluoride in the effluent. Tests 14 and 16 present somewhat puzzling results; in both cases space velocity and dilution were decreased from optimum conditions, and a small amount of nitrogen trifluoride passed through the bed. In spite of the anamolous results, the nitrogen trifluoride conversion to innocuous products was promising under all conditions tested.

Under all conditions used in testing with the charcoal beds, there was no indication of N_2F_4 forming although there was a slight increase in the concentration of active fluoride species as evidenced by the exit gas active fluoride analyses.

Although no bed agglomeration occurred, localized reaction zones also caused a slight problem with activated charcoal bed reactions. The high temperatures attained in these zones caused the formation of metal fluorides at reactor surfaces. Material balances from inlet and outlet gas streams shown in Table 2.8-2 suggest this side reaction. Nitrogen balances were generally within 10%, while fluorine content of the exit gas stream was consistently low. Possibly a significant portion of the fluorine is retained in the bed as solid carbon-fluorine addition compounds and as adsorbed fluorocarbons on the charcoal. Some metal fluorides were found interspersed with bed material upon post-reaction examination of beds. The occurrence of this reaction indicates a need for proper reactor design considerations including increased bed diameters and suitable materials of construction.

In summary, limestone proved to be unsatisfactory as a bed material in static bed NF_3 waste disposal applications due to its tendency to agglomerate in the localized high temperature zones characteristic of the reaction. Preheated activated charcoal was found to be a suitable bed material for converting nitrogen trifluoride to innocuous compounds because in this application not only because the bed particles do not agglomerate, but the conversion of nitrogen trifluoride to innocuous compounds is accomplished with negligible production of gaseous active fluoride species.

TABLE 2.8-2

NITROGEN AND FLUORINE MATERIAL BALANCES IN WASTE DISPOSAL TEST GAS STREAMS

	Nitrogen				Fluoride			
	In		Out		In		Out	
Test #	(SCFM* x 10^2)	millimoles per sec	(SCFM* x 10^2)	millimoles per sec	(SCFM x 10^2)	millimoles per sec	(SCFM x 10^2)	millimoles per sec
14	59.6	11.6	62.1	12.1	14.0	2.74	0.92	0.18
15	90.2	17.6	101.5	19.8	14.0	2.74	2.51	0.49
17	124.1	24.3	119.0	23.3	18.5	3.62	5.73	1.12
18	99.7	19.5	108.2	21.1	18.5	3.62	2.33	0.46
21	62.0	12.1	94.2	18.4	12.8	2.50	5.01	0.98

*SCFM at 70°F

2.0,

2.9 NITROGEN TRIFLUORIDE ANALYSES AND COMPRESSIBILITY FACTORS

2.9.1 Nitrogen Trifluoride Analyses

The analyses of the nitrogen trifluoride were conducted according to the procedures described in AFRPL-TR-76-89 "Nitrogen Trifluoride Analytical Procedures Final Report" by L. A. Dee and W. T. Leyden. The analyses conducted after testing are reported in the appropriate sections containing the test data. The analysis of the as-received nitrogen trifluoride is reported in Table 2.9-1.

2.9.2 Compressibility Factors for Gaseous Nitrogen Trifluoride

A review of the literature indicated that no experimental compressibility data were available at 344 K (160 F) and at pressures as high as 17.24 MN/m^2 (2500 psia) for gaseous nitrogen trifluoride. In order to assure safe operations during the high pressure testing reported in Sections 2.2, 2.3, and 2.6, experimental determinations of the compressibility factors were made in the temperature range from 273 to 344 K (32 to 160 F) and a pressure range of 3.45 to 20.7 MN/m^2 (500 to 3000 psia).

A series of tests were conducted in which various quantities of NF_3 were placed in fixed volume stainless steel apparatuses which were completely immersed in water baths at various temperatures. The bath temperatures were measured with thermometers and the pressures were measured with a 3000 psia Taber transducer which is accurate to within 0.5%. The data are tabulated in Table 2.9-2. Selected data are plotted in Figure 2.9.1. Included in the plots are a few points calculated from the compressibility tables in R. C. Reid and T. K. Sherwood, "Properties of Gases and Liquids", 2nd Edition, McGraw-Hill, 1966. The agreement between the experimentally determined compressibility factors and the calculated values is excellent.

TABLE 2.9-1

CHEMICAL ANALYSIS OF THE AS-RECEIVED NITROGEN TRIFLUORIDE

Cylinder No.	Composition, Weight Percent NF_3	Active Fluoride as HF	N_2	CO/O_2	CF_4	CO_2	N_2O
17228-C	98.68	0.17	0.20	0.10	0.75	0.013	0.083
17319-C	98.72	0.10	0.13	0.45	0.51	0.016	0.070
17341-C	98.3	0.077	0.31	0.74	0.38	0.03	0.013
H 81136	99.56	0.0001	0	0.35	0.009	0	0.074
H 55957	98.68	0.0002	0	0.24	1.03	0.011	0.048
H 79957	99.01	<0.0001	0	0.27	0.65	0.012	0.053
N 36777	99.66	0.0006	0	0.29	0.017	0.011	0.017
P 178684	99.68	0.0003	0	0.29	0.017	0	0.014
309029	99.58	<0.0001	0.039	0.30	0.030	0.008	0.043
237250	98.76	0.0017	0.22	0.31	0.67	~0	0.041
C 5298	99.64	0.016	~0	0.31	0.014	~0	0.025
385580	99.50	0.0027	0.12	0.31	0.015	0.027	0.023

TABLE 2.9-2

DATA INDICATIVE OF THE VARIATION OF THE COMPRESSIBILITY FACTORS OF GASEOUS NITROGEN TRIFLUORIDE AS A FUNCTION OF TEMPERATURE AND PRESSURE

Specific Volume cc/gm	Temperature °K	Temperature °C	Pressure MN/m^2	Pressure psia	Compressibility Factor, Z
8.34	273.2	0	3.23	469	0.843
8.34	294.3	21.1	3.55	515	0.859
8.34	321.2	48.0	4.03	584	0.895
8.34	344.2	71.0	4.38	635	0.909
3.75	273.2	0	5.89	854	0.690
3.75	292.9	19.7	6.72	974	0.734
3.75	324.3	51.1	8.09	1174	0.800
3.15	273.2	0	6.58	954	0.648
3.15	295.2	22	7.69	1115	0.700
3.15	323.6	50.4	9.27	1345	0.772
3.15	344.2	71.0	10.32	1497	0.809
2.22	273.2	0	8.09	1174	0.560
2.22	291.6	18.4	9.63	1397	0.625
2.22	318.7	45.5	12.02	1744	0.715
2.22	344.3	71.1	14.24	2065	0.785
2.15	326.2	53	12.86	1865	0.724
2.15	344.3	71.1	14.55	2111	0.778
1.60	273.2	0	10.16	1474	0.509
1.60	293.5	19.3	12.82	1860	0.598
1.60	310.2	37	15.44	2239	0.681
1.60	316.7	43.5	16.33	2369	0.706
1.60	344.3	71.1	20.23	2934	0.806
1.21	273.2	0	13.89	2015	0.527
1.21	283.5	10.3	16.20	2350	0.593
1.21	288.2	15.0	17.26	2504	0.622
1.21	293.2	20.0	18.37	2664	0.650
4.75*	273.2	0	4.99	724	0.740
4.75*	295.2	22	5.70	826	0.781
4.75*	324.7	51.5	6.72	974	0.839
4.75*	344.3	71.1	7.27	1054	0.857
3.21*	273.2	0	6.58	954	0.660
3.21*	292.2	19.0	7.61	1104	0.715
3.21*	324.0	50.8	9.34	1354	0.792
3.21*	344.2	71.0	10.37	1504	0.829

*Volume of system was 2.465 times greater than initial system

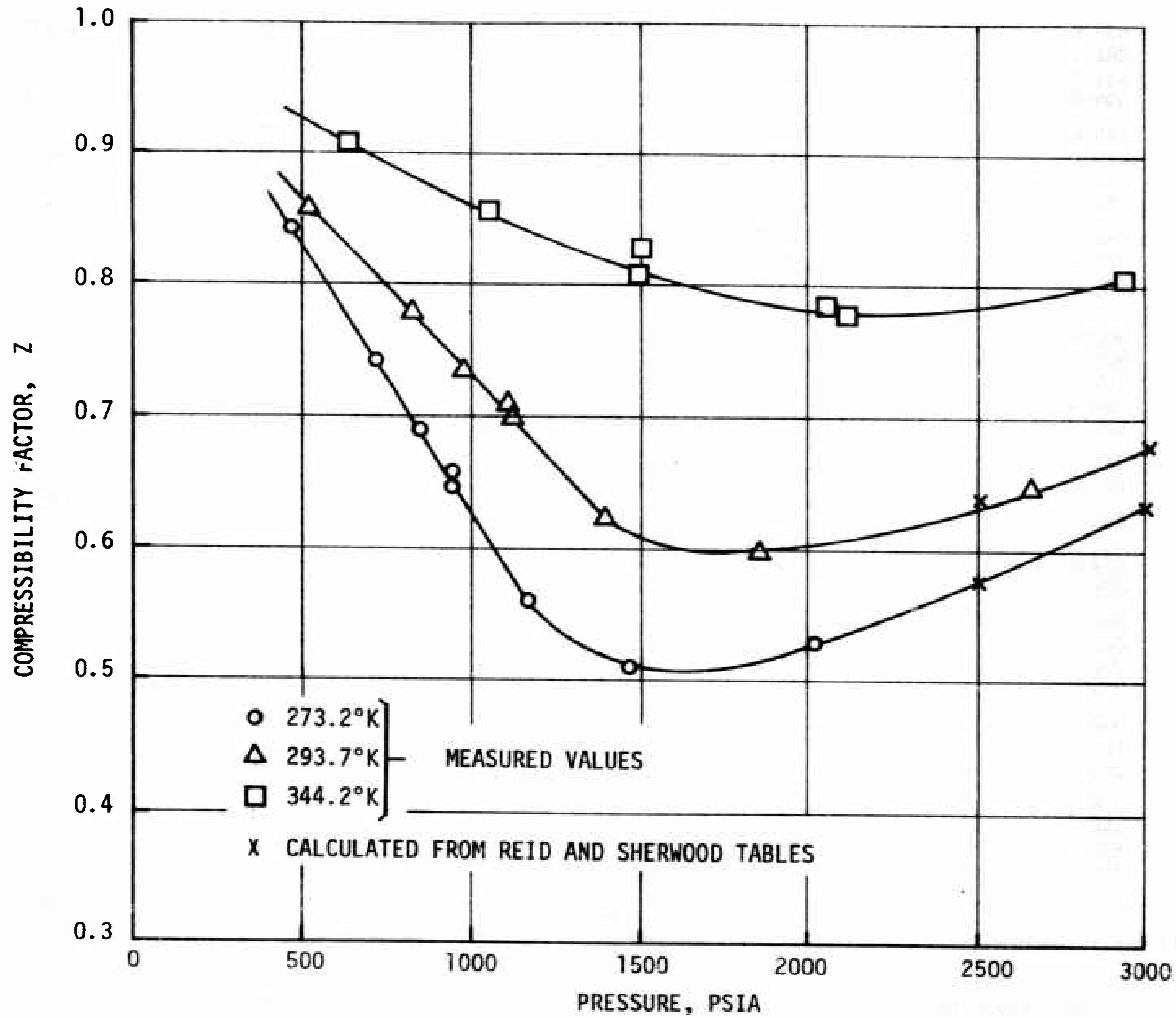


Figure 2.9.1. The Compressibility Factor for Nitrogen Trifluoride at Various Temperatures Versus Pressure

2.0, Experiment Results and Discussion (cont.)

2.10 WATER HAMMER TESTS WITH NON-METALLIC MATERIALS

The objective of the water hammer tests was to determine the effects of a shock wave promulgated through liquid nitrogen trifluoride on non-metallic materials. The non-metal candidates which were considered appropriate for this test are:

Polytetrafluoroethylene
Carbon (CJPS)
Kel-F 81
Silastic LS-53
Viton
Polyethylene

The test apparatus, procedures and results are discussed below.

2.10.1 Test Method and Apparatus

The apparatus used was the U-tube adiabatic compression test apparatus which was modified to incorporate a means of temperature-conditioning the U-tube to condense the nitrogen trifluoride as liquid. A schematic diagram of the entire appratus for handling the nitrogen trifluoride and conducting the test is shown in Figure 2.5.1. The schematic diagram of the U-tube adiabatic compression apparatus is shown in Figure 2.10.1; a photograph of the apparatus is shown in Figure 2.10.2; and the schematic of the test specimen holder with the test specimen in place is shown in Figure 2.10.3. The test specimen holder was 6.35 mm (.25 in.) hollow AN plug used to seal the end of the U-tube. The test specimen was a strip of material 2.5 mm (0.10-in.) long which was wedged into the end of the hollow AN plug and cemented in place with Sauereisen. The U-tube was fabricated from Hastelloy-X 6.35 mm (.25 in.) tubing approximately 40.6 cm (16-in.) long.

The tests were conducted in the following manner. The U-tube was attached to the apparatus and the specimen holder used to seal the open-end of the U-tube. The tube was then evacuated to 133 N/m^2 (1 torr) or less, a predetermined quantity of NF_3 was gradually introduced into the assembly to a predefined pressure level and condensed in liquid nitrogen. The pneumatic remotely-operated valve was then actuated and the nitrogen from the accumulator tank was used to drive the liquid NF_3 against the non-metal specimen. The U-tube assembly was then vented and flushed with nitrogen and the test specimen was examined visually to ascertain if any attack occurred. Microscopic examination was used to evaluate the samples which were not totally consumed in the test. A 1000 lb burst disc

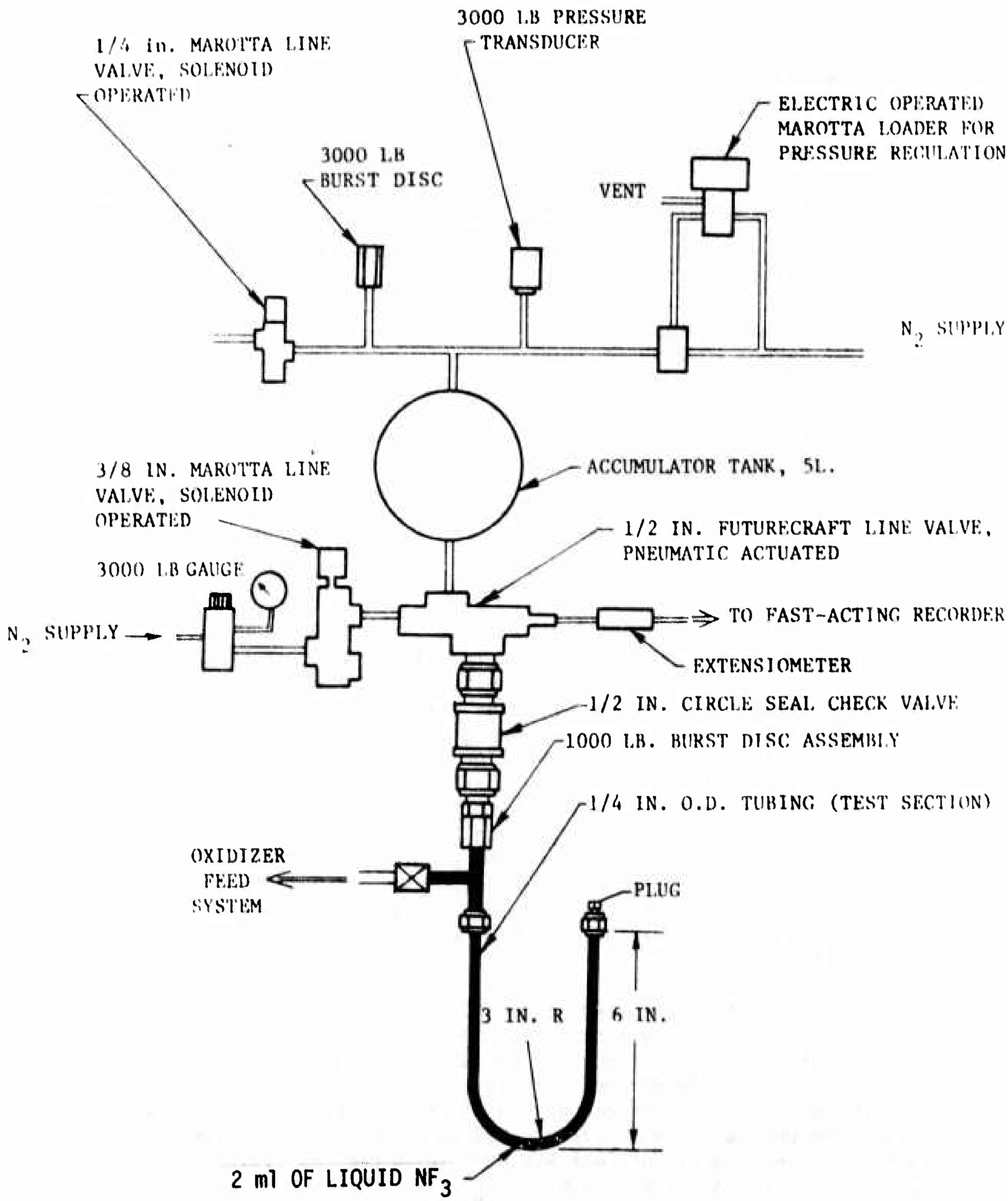


Figure 2.10.1. Schematic Diagram of U-Tube Adiabatic Compression Apparatus as Used in Water Hammer Tests

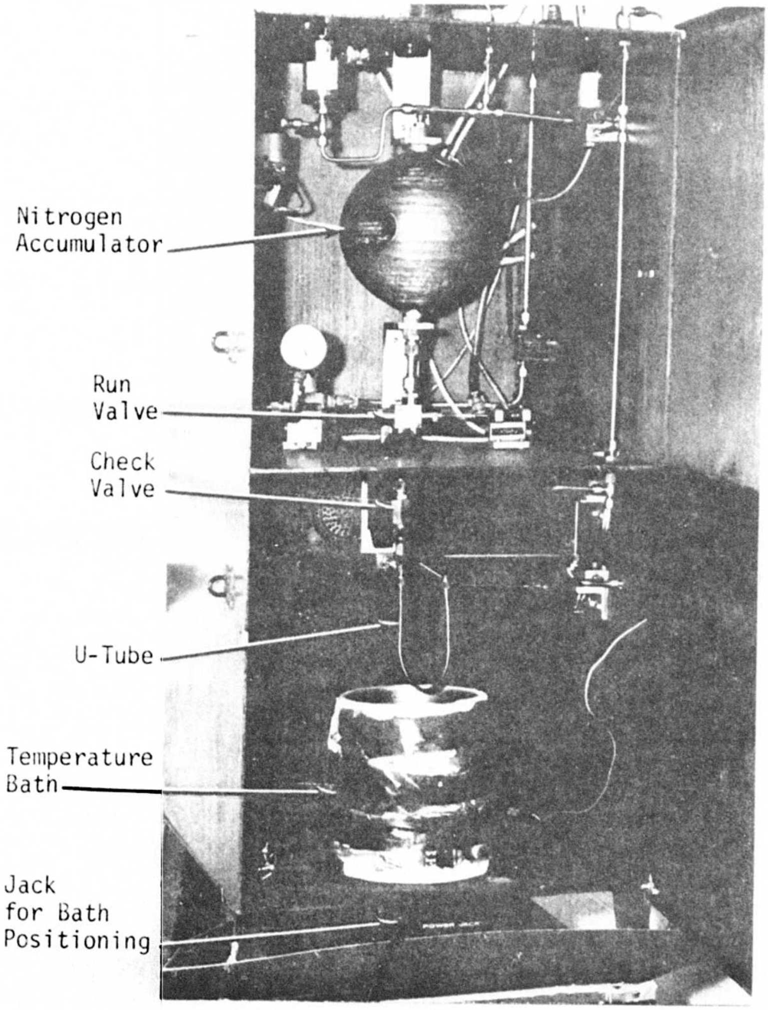


Figure 2.10.2. Photograph of Apparatus Used in Water Hammer Tests

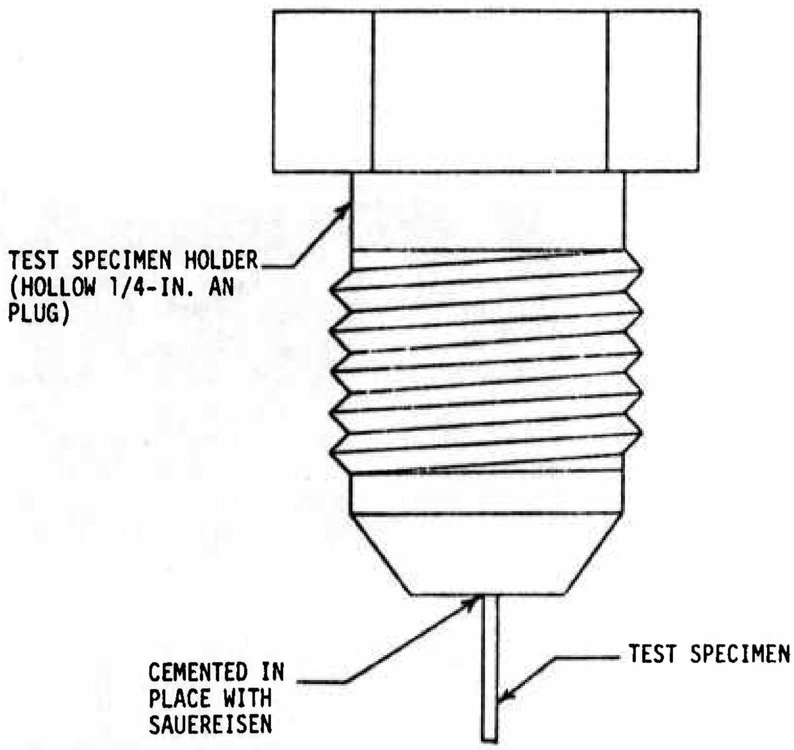


Figure 2.10.3. Schematic of Test Specimen Holder with Test Specimen in Place

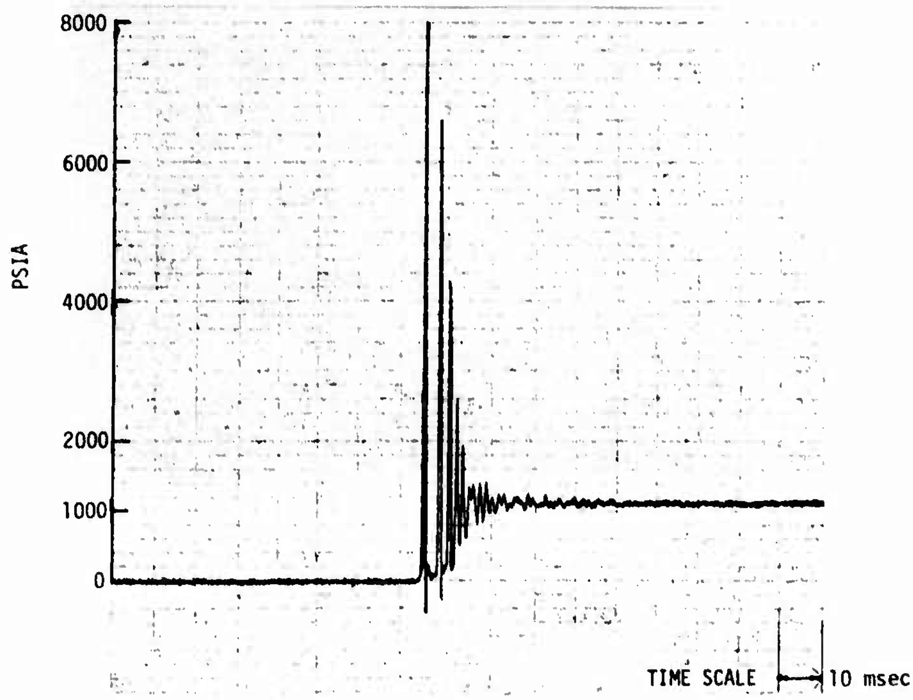


Figure 2.10.4. Pressure Trace of Water Hammer Effect Using a Driving Pressure of 7.69 MN/m^2 (1100 psig) and with Liquid Water in the U-Tube

made of 304-L stainless was used in each test to seal the pneumatic valve and check valve assembly from the nitrogen trifluoride atmosphere prior to the test. At driving pressures below 7.58 MN/m^2 (1100 psia) no burst disc is used. The driving pressure in the accumulator tank is varied with each material to achieve final pressures at which the non-metals are susceptible to attack. The test specimen is replaced after each test to insure that comparable surfaces are being exposed to the test conditions. The pneumatic valve opens completely within 1.5 milliseconds so with an accumulator pressure of 6.89 MN/m^2 (1000 psia), the minimum pressurization is 4.6 GN/m^2/sec (6.7 x 10^5 psi/sec). An example of the water hammer effect achieved with the apparatus is shown in Figure 2.10.4. The driving pressure of 7.69 MN/m^2 (1115 psia) produced a pressure spike of 55.2 MN/m^2 (8000 psia), greater than a seven-fold increase over the driving pressure.

2.10.2 Experimental Results

The data obtained from the water hammer test are presented in Table 2.10-1.

The significant items to note from the data are as follows. The Kel-F 81 is apparently the most durable and compatible material evaluated in this test. Teflon ranks second; although no chemical incompatibility of the Carbon (CJPS) with NF_3 was apparent, the structural integrity of the carbon specimens was destroyed at the higher driving pressures. Viton undergoes small changes in its surface at driving pressures above 6.3 MN/m^2 (915 psia). Both polyethylene and Silastic LS 53 ignited at the higher driving pressures but polyethylene withstood higher driving pressure without reaction than the Silastic LS 53.

TABLE 2.10-1

BEHAVIOR OF VARIOUS NON-METALS SUBJECTED TO A SHOCK WAVE IN LIQUID NITROGEN TRIFLUORIDE

Non-Metal	Initial Liquid Temperature °K	°F	Driving Pressure MN/m^2	psia	Results
Viton	77	-321	13.9	2015	Slight change in edge
			13.9	2015	Some surface change
			12.5	1815	Some surface change
			10.4	1515	Some surface change
			8.38	1215	Some surface change
			7.00	1016	Very slight surface change
			7.00	1015	Slight surface change
			6.31	915	No effect
			6.31	915	No effect
			6.31	915	No effect
Polyethylene	77	-321	13.9	2015	Surfaced burned
			10.4	1515	Surfaced burned
			8.38	1215	Surfaced burned
			7.69	1115	Surfaced burned
			7.69	1115	Surfaced burned
			7.00	1015	No effect
Carbon (CJPS)	77	-321	13.9	2015	Sample crumbled
			12.5	1815	Sample crumbled
			11.8	1715	Sample crumbled
			11.1	1615	Sample crumbled
			10.4	1515	No effect
			7.00	1015	No effect
Silastic LS 53	77	-321	13.9	2015	Sample burned
			10.4	1515	Sample burned
			7.00	1015	Sample burned
			7.00	1015	Sample burned
			3.55	515	Slight surface change
			2.86	415	No effect
Kel-F 81 CTFE	77	-321	17.3	2515	No effect
			17.3	2515	No effect
			13.9	2015	No effect
			13.9	2015	No effect
Polytetra fluoroethylene	77	-321	17.3	2515	Slight surface change
			17.3	2515	No effect
			17.3	2515	No effect
			17.3	2515	Sample burned
			16.7	2415	No effect
			16.7	2415	No effect
			13.9	2015	No effect
			13.9	2015	No effect

2.0, Experiment Results and Discussion (cont.)

2.11 NATURE AND RATE OF FORMATION OF PASSIVATION FILMS

The purpose of this task was to determine whether a stoichiometric fluoride compound formed as a passive film on metals in the temperature range from 195 to 344 K and to determine the rate at which such a film would form.

2.11.1 Apparatus and Procedures

The nature and rate of formation of passivation films in liquid and gaseous nitrogen trifluoride were evaluated by varying exposure times of selected metals to nitrogen trifluoride, and analyzing the resulting surfaces for the formation of surface films utilizing Ion Scattering Spectrometery (ISS) coupled with a quadrupole mass analyzer which allows analysis of the secondary ion mass (SIMS) values and residual gas analysis (RGA). The data obtained from the procedure allows identification of the chemical elements present as a function of their distance from the surface. The result is a layer by layer examination of the sample. The analyses were conducted by SEAL, Inc.* The representative metals were subjected to gaseous nitrogen trifluoride exposure at 344 K (160 F) and 3.45 MN/m^2 (500 psia) for periods of 30 seconds, 30 minutes, 30 hours and 30 days; the same metals were subjected to liquid nitrogen trifluoride exposure at 195 K (-78 C) for periods of 30 minutes, 30 hours, and 30 days. The metals used in the evaluation were: 2219 Aluminum, 304 Stainless Steel, and Nickel 200.

Prior to exposure to the nitrogen trifluoride, the metal samples were washed with a detergent solution (Turco Plaudit) and then washed with isopropanol and vacuum dried. They were not exposed to any pickling solutions and therefore any fluoride present in the specimens must be obtained from the nitrogen trifluoride.

The elemental peak heights in the ISS spectra were converted into concentration values in atomic percent in the following manner. First, the peak areas were measured in each spectra; second, because only the oxide or fluoride anions were present in the spectra, the ionic radii of the oxide and fluoride were chosen as representative of the size of the anions; third, the relative number of atoms of each species on the surface was calculated by dividing the areas under each peak by the square of the corresponding ionic radii; and fourth, the atomic percent was calculated from the relative number of atoms of each species present.

*Scanning Electron Analysis Laboratories, Inc., Los Angeles, CA 90066.

2.11, Nature and Rate of Formation of Passivation Films

The elemental concentrations, in atomic percent, for fluorine and oxygen were then correlated with the fluorine and oxygen peak heights obtained from the SIMS spectra at the same sputtered depth for each sample. The correlation between the ISS atomic percent concentration and the SIMS peak heights exhibited appreciable scatter, but it was deemed acceptable by the analyst. Correlation lines for converting SIMS peak heights to atomic percent were drawn through the origin; factors were obtained from the slopes of these lines to permit conversion of SIMS peak heights to atomic percent. Using these conversion factors, the fluorine and oxygen peak heights obtained as a function of depth for each sample were converted to atomic percent.

There was no evidence of nitrogen or fluorides adsorbed on the surface of the materials, only a hydrocarbon which is attributable to the isopropanol.

Because the metal specimens were analyzed as two batches, two different correlation sets were used to calculate the concentrations; one correlation set was used for metal specimens exposed for 30 days and 30 hours to nitrogen trifluoride and the other set was used for metal specimens exposed for 30 minutes and 30 seconds to nitrogen trifluoride.

2.11.2 Test Results

The data obtained from the 304 stainless steel specimens are shown in Figures 2.11.1 and 2.11.2; the data obtained from the nickel 200 specimens are presented in Figures 2.11.3 and 2.11.4; and the data obtained from the aluminum 2219 specimens are shown in Figures 2.11.5 and 2.11.6.

If the surface were converted to a pure layer of stoichiometric compounds, the fluoride concentration should be a minimum of 66 atomic percent for the 304 stainless steel, 66 atomic percent for the Nickel 200, and 75 atomic percent for the 2219 Al. From the data presented, it is apparent that none of the materials evaluated form such a layer at the surface, because the maximum fluoride concentration detected is 11 atomic percent near the surface of the 304 stainless steel.

For the 304 stainless steel it is apparent that the diffusion of the fluoride into the surface is enhanced significantly by exposure at 344 K (160 F) and 3.45 MN/m^2 (500 psia) to gaseous nitrogen trifluoride as compared to exposures in liquid nitrogen trifluoride at 195 K (-78 C). After 30 days in liquid nitrogen trifluoride at 195 K (-78 C) less than 1 atomic percent fluoride is present on the surface of the metal,

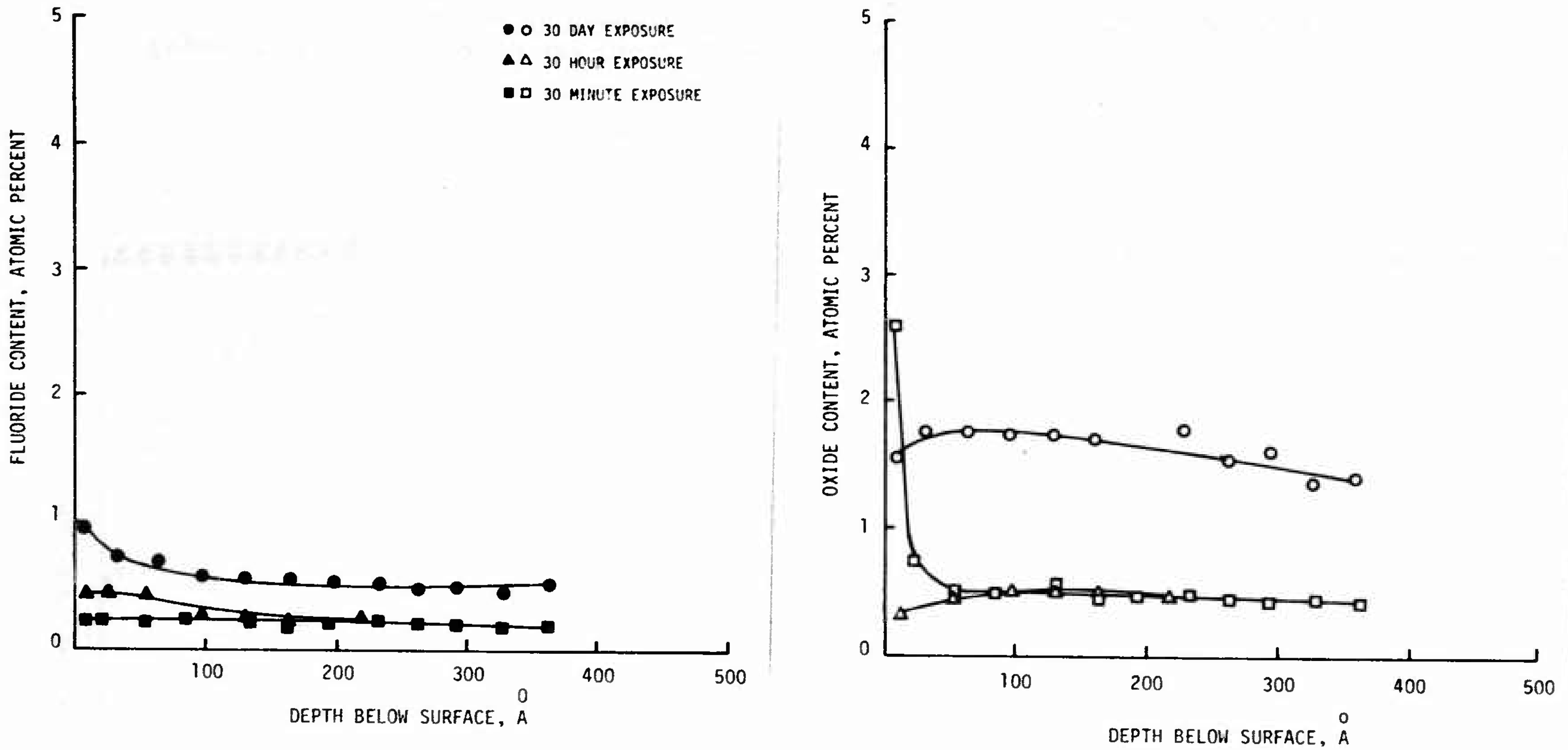


Figure 2.11.1. Fluoride and Oxide Content of 304L Stainless Steel Surfaces Exposed to Liquid NF_3 at 195K (-78C)

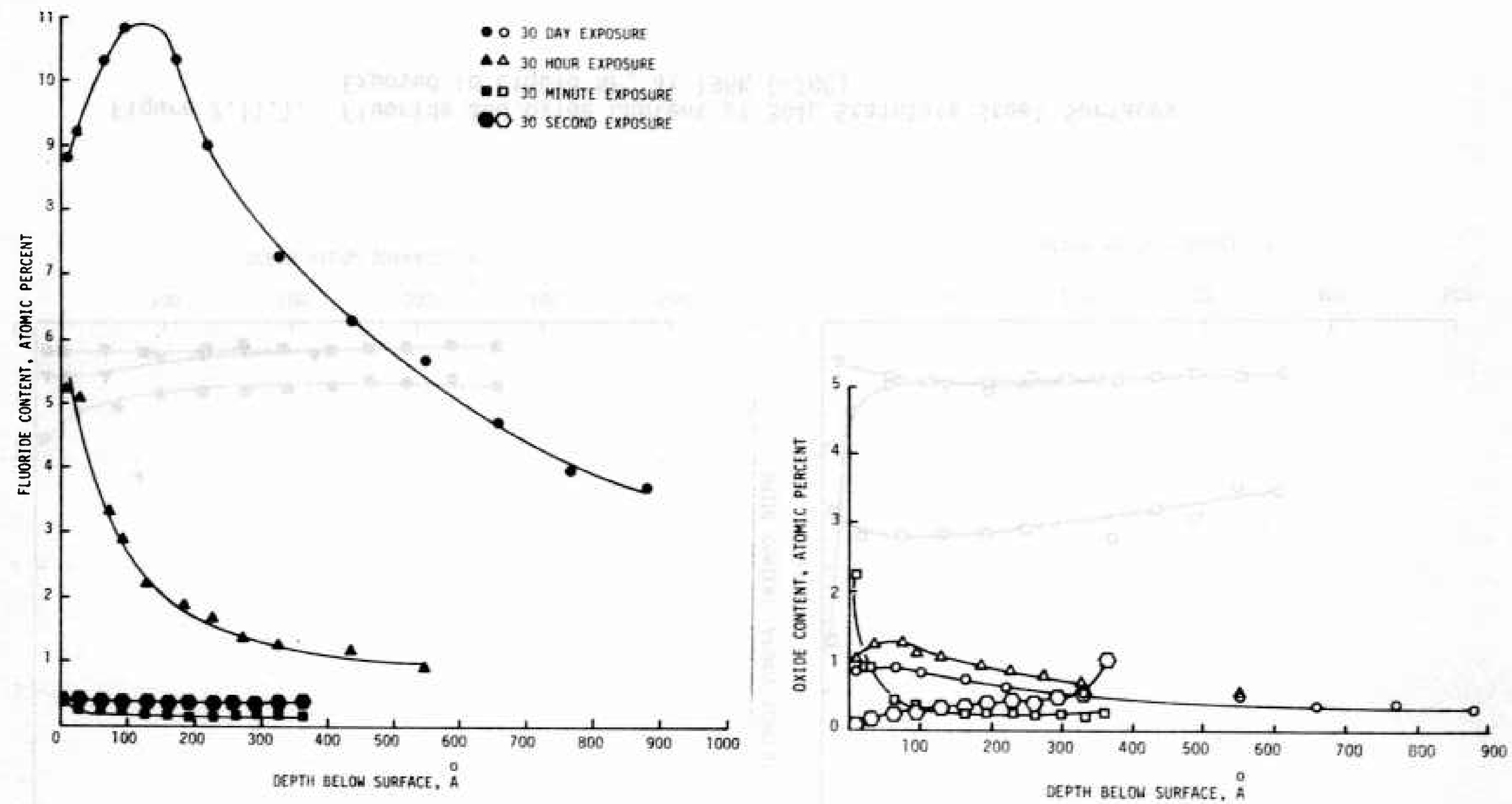


Figure 2.11.2. Fluoride and Oxide Content of 304L Stainless Steel Surfaces Exposed to Gaseous NF_3 at 344K (160F) and 3.45 MN/m^2 (500 psia)

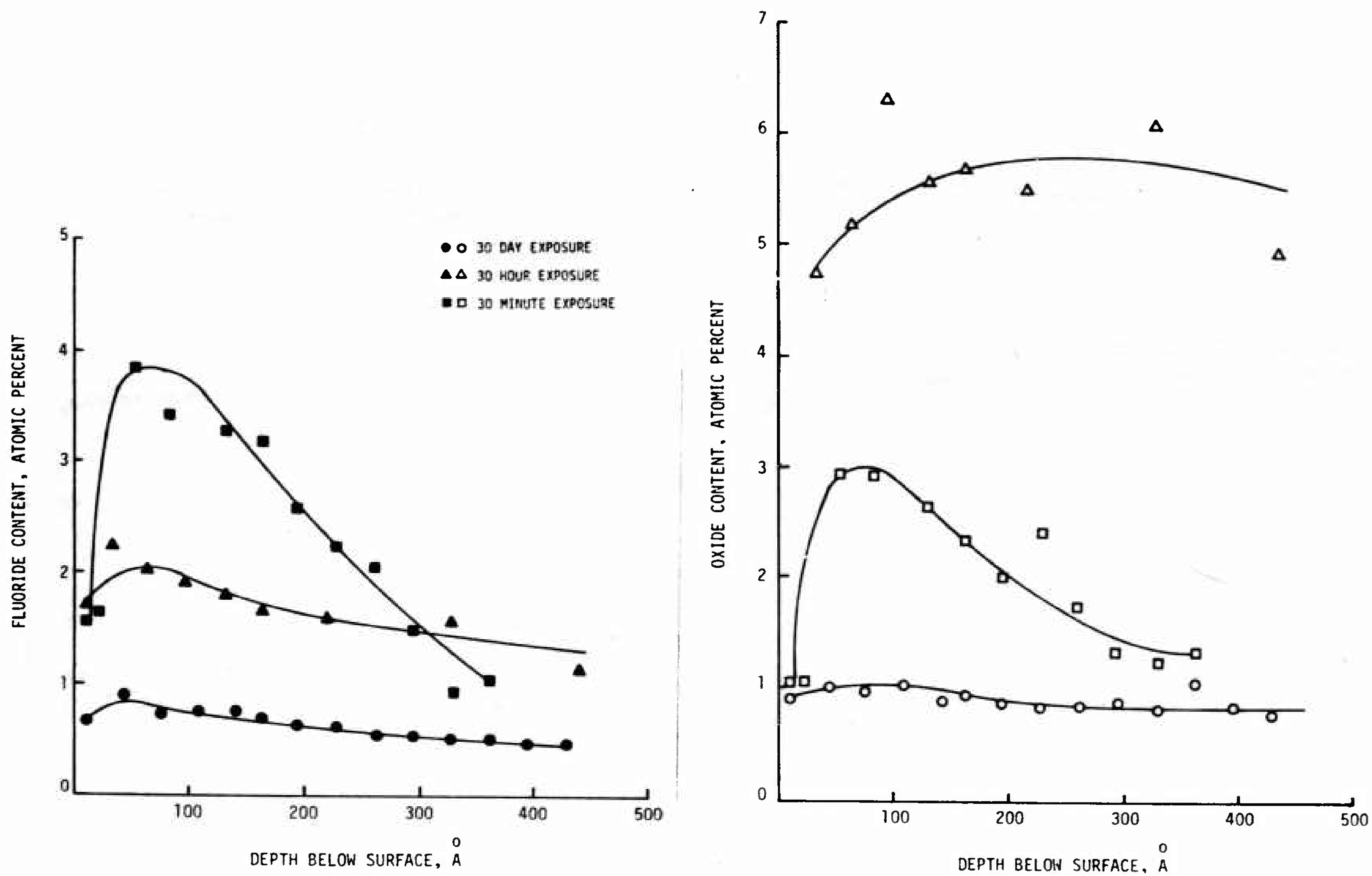


Figure 2.11.3. Fluoride and Oxide Content of Nickel 200 Surfaces Exposed to Liquid NF_3 at 195K (-78C)

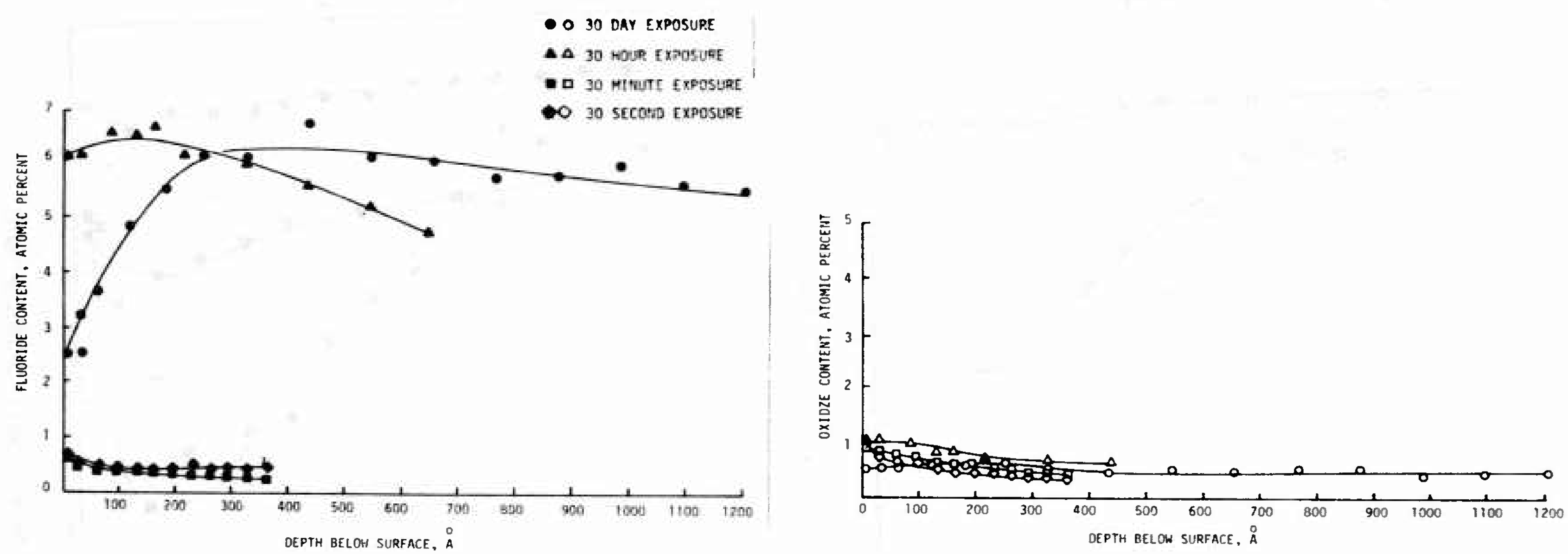


Figure 2.11.4. Fluoride and Oxide Content of Nickel 200 Surfaces Exposed to Gaseous NF_3 at 344K (160F) and 3.45 MN/m^2 (500 psia)

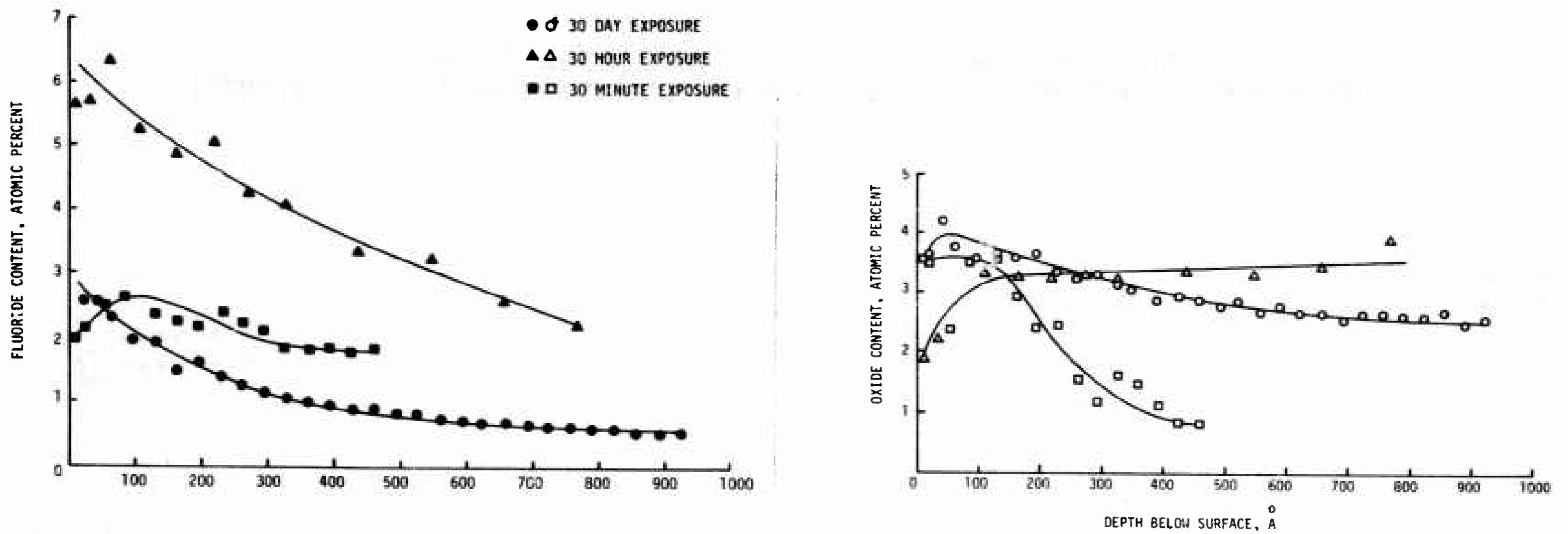


Figure 2.11.5. Fluoride and Oxide Content of 2219 Aluminum Surfaces Exposed to Liquid NF_3 at 195K (-78C)

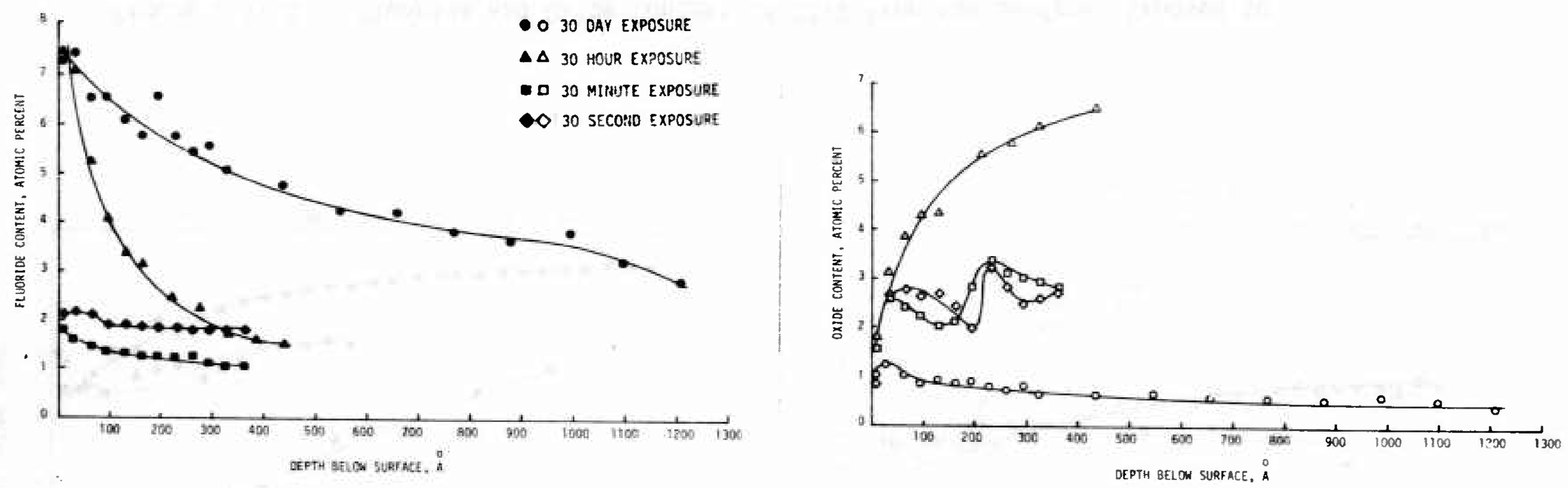


Figure 2.11.6. Fluoride and Oxide Content of 2219 Aluminum Surfaces Exposed to Gaseous NF_3 at 344K (160F) and 3.45 MN/m^2 (500 psia)

while after 30 days in gaseous nitrogen trifluoride at 3.45 MN/m^2 (500 psia) and 344 K (160 F), 11 atomic percent fluoride is apparently present. The data also indicate that the penetration of the metal surface by the fluoride is a relatively slow process at 344 K (160 F). Several hours elapse before any appreciable concentration is detectable below the immediate surface; after 30 second and 30 minute exposure the concentration level is less than 0.5 atomic percent which considering the semi-quantitative nature of the data is detectable but not significant. In liquid NF_3 at 195 K (-78 C), the fluoride penetration rate is extremely low, with less than 0.6 atomic percent of fluoride present after 30 days exposure. No nitrogen species were detected on or below the surface of the 304 stainless steel in either the liquid or gaseous exposure tests.

For Nickel 200, much the same situation exists as that which occurs with the 304 stainless steel at 344 K (160 F). Fluoride penetration is enhanced by the higher temperature; no significant fluoride penetration of the surface occurs within 30 minutes in gaseous NF_3 at 344 K (160 F); and it appears that 400 Å below the surface a maximum concentration of 6.8 atomic percent fluoride is present after 30 days in gaseous NF_3 at 344 K (160 F). The data from the liquid nitrogen trifluoride exposure tests are completely reversed from what reason would predict. The highest fluoride concentration is produced by the shortest exposure period, 30 minutes; and the lowest fluoride concentration is produced by the longest period of exposure, 30 days. The anomaly exists, and perhaps is a reflection on the semi-quantitative nature of the analytical method. The data for the 30 hour and 30 day exposed samples were calculated with one set of calibration curves, and the data for the 30 minute exposed was calculated with the other set of calibration curves. In spite of this dilemma, it is apparent that at 300 Å below the nickel 200 surface, the fluoride concentration may be detectable, but is not significant.

For the 2219 aluminum samples exposed to the gaseous nitrogen trifluoride at 344 K (160 K) and 3.45 MN/m^2 (500 psia) it appears that several hours are required before significant quantities (5 atomic percent) of fluoride penetrate the metal surface; the reversal of the 30-second and 30-minute exposure data may again be due to the semi-quantitative characteristic of the data. The data obtained from the samples exposed to the liquid nitrogen trifluoride are confusing with regard to rate of fluoride penetration because the 30-hour values are nearly three times as large as the 30 day data. But the data do indicate that a pure aluminum fluoride passivation film does not exist on the metal surface.

2.12 SOLUBILITY OF PASSIVATION FILMS IN LIQUID NITROGEN TRIFLUORIDE

This study was contingent on the results obtained in the previous study. If there was a distinct difference in the passivation films formed in liquid and gaseous nitrogen trifluoride, then an evaluation of the solubility of such films was warranted. The results indicated that at the conditions used in the tests no distinct passivation films formed, but the data did indicate that after 30 days exposure to gaseous nitrogen trifluoride at 344 K (160 F) and 3.45 MN/m^2 (500 psia), the fluoride concentration levels in the metal surfaces were substantially greater than after 30 days exposure in liquid nitrogen trifluoride at 195 K (-78 C). After 30 days the maximum fluoride concentrations near the metal surfaces from liquid and gaseous exposures were as follows: Ni 200, < 1 versus 6.7 atomic percent; Al 2219, 2.5 versus 7.4 atomic percent; and 304 stainless steel, < 1 versus 10.8 atomic percent. Whether this behavior is strictly a function of temperature or whether the presence of the liquid phase affects the resultant concentration levels was not uniquely resolved.

2.12.1 Apparatus and Procedures

Tests were conducted in which samples of Ni 200, 304 stainless steel, and 2219 aluminum were initially subjected to a 30 day exposure in gaseous nitrogen trifluoride at 344 K (160 F) and 3.45 MN/m^2 (500 psia) and then the samples were subsequently exposed to liquid nitrogen trifluoride for 30 days at 195 K (-78 C). After the second exposure period the metal surfaces were subjected to the SIMS and ISS analyses described in Section 2.11.1.

2.12.2 Test Results

The data obtained from the tests is presented in Figure 2.12.1. The data indicate that the maximum fluoride concentration near the metal surface is as follows: Ni 200, 4.9 atomic percent; 2219 Al, 2.5 atomic percent; 304 stainless steel, 2.5 atomic percent.

The implications from the data are: (1) that the fluoride present in the Nickel 200 from gaseous exposure is not affected by later exposure to liquid nitrogen trifluoride (the 4.9 versus the 6.7 atomic percent value measured for a sample exposed to gaseous nitrogen trifluoride only are in agreement with each other as one considers the semi-quantitative nature of the data), and (2) the fluoride present in the 2219 aluminum and 304 stainless samples from exposure to gaseous trifluoride may have been removed to some extent. The apparent reductions from 7.4 to 2.5 atomic percent in 2219 aluminum and from 10.8 to 2.5 atomic percent in 304 stainless steel are large enough to establish some validity to the actual removal of

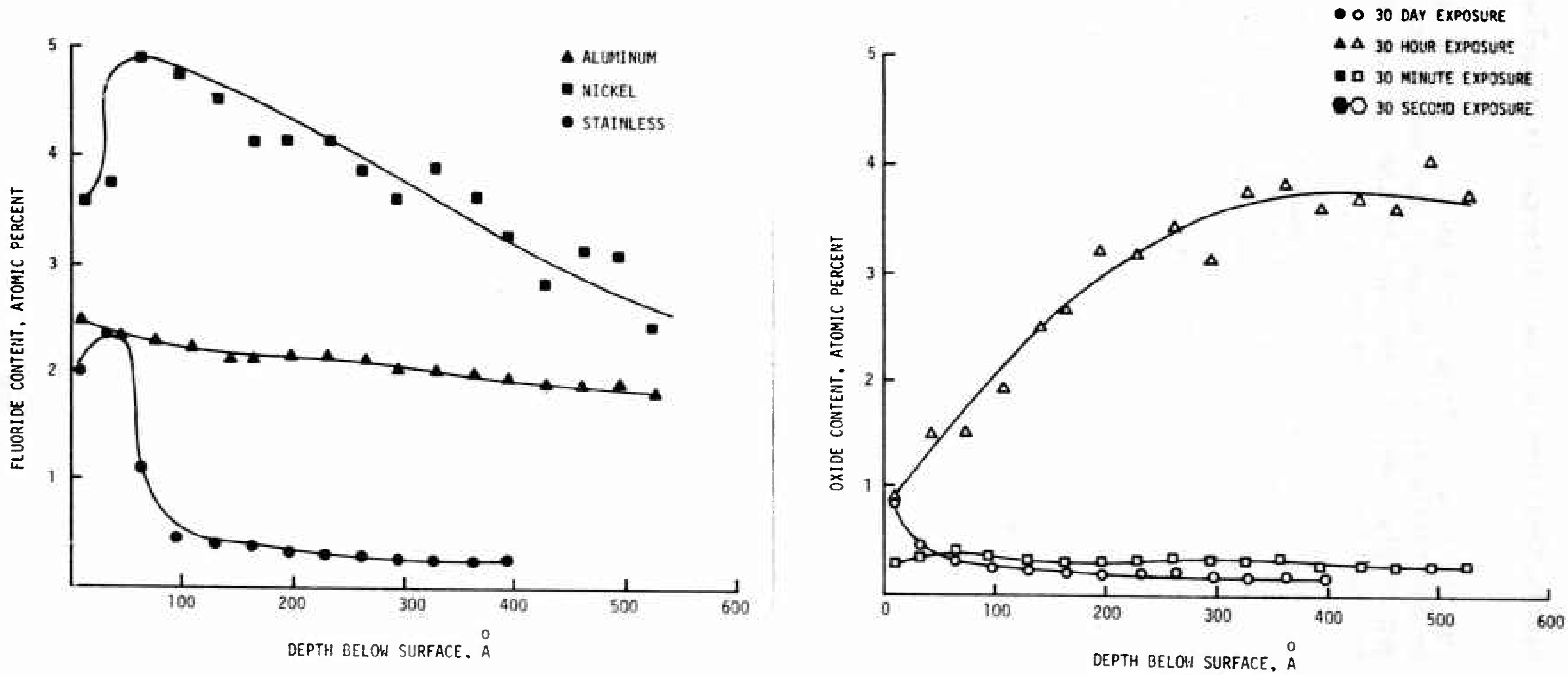


Figure 2.12.1. Fluoride and Oxide Content of Metal Surfaces Exposed to Gaseous NF_3 for 30 Days at 344K (160F) and 3.45 MN/m^2 (500 psia) Followed by 30 Days of Immersion in Liquid NF_3 at 195K (-78C)

some of the fluoride by the liquid nitrogen trifluoride. In any case, very little fluoride is present initially near the metal surfaces after exposure to the gaseous nitrogen trifluoride, so the quantity that can be removed by the presence of the liquid phase is also small.

Analysis of the wash water used to flush any residuals after the evaporation of the nitrogen trifluoride from the sample container indicated that no Al, Fe, Cr, and Ni compounds were present in the nitrogen trifluoride. The analyses were conducted by atomic absorption spectroscopy and limits of detection are 1 ppm for aluminum and 0.2 ppm for iron, chromium, and nickel, and correspond to microgram quantities of the metals. The findings are consistent with the fact that very little fluoride is present near the metal surfaces to form compounds with the base metals.

In summation, there is some evidence that the fluorides near the surface of 304 stainless steel and 2219 aluminum may be removed by the presence of liquid nitrogen trifluoride, but the fluorides are not apparently removed from Nickel 200. The data from the static exposure tests reported in Section 2.2 indicates that none of the alloys used in this study are subject to corrosion in liquid nitrogen trifluoride.

2.13 EFFECTS OF CONTAMINANTS ON METAL COMPATIBILITY IN NITROGEN TRIFLUORIDE

The purpose of the tests reported herein was to establish the effect of common contaminants on the chemical compatibility of nitrogen trifluoride with various materials. The effects of five contaminants, (1) fingerprints, (2) petroleum jelly, (3) lightweight machine oil, (4) brazing flux, and (5) a fluorocarbon oil, FC-75, were evaluated in the tests described below.

2.13.1 Apparatus and Procedures

Three types of tests were conducted with the candidate contaminants; (1) screening tests, (2) adiabatic compression tests, and (3) gaseous flow tests. The apparatus and procedure for each type of test is discussed.

2.13.1.1 Screening Tests

The screening tests were conducted with the apparatus described in Section 2.1.2.3 and shown in Figures 2.1.4 and 2.1.5. The tests were conducted in the following manner. The candidate contaminants were placed in a small aluminum cup except for the fingerprint contamination which was placed on Silastic LS-53 specimens to determine whether the contaminant would lower the threshold temperature at which the Silastic LS-53 previously had exhibited a slight endotherm in the presence of nitrogen trifluoride. The specimens were heated from ambient temperature to 478 K (400 F) in a period of 10 to 15 minutes while either oxygen or nitrogen trifluoride was flowing at a rate of 60 ml/min onto the surface of the specimens. In addition to the thermal measurements, visual changes in the specimens were noted. The total pressure in the reaction flask was one atmosphere.

2.13.1.2 Adiabatic Compression Tests

The adiabatic compression tests were conducted with the apparatus and procedures described in Section 2.5.1. The tests were conducted by placing small quantities of the contaminants on nickel 200 and 347 stainless steel specimens which were subjected to adiabatic compression in gaseous nitrogen trifluoride. The metal specimens were examined prior to testing and after testing microscopically to establish the reactive threshold levels.

2.13.1.3 Gaseous Flow Tests

The gaseous flow test appratus is shown in Figures 2.4.1, 2.4.2 and 2.4.5. The metal specimens were contaminated by applying

the contaminants to the test orifice specimen with a cotton swab. The test procedures and data evaluation process are described in Section 2.4.1.

2.13.2 Experimental Results

2.13.2.1 Screening Test Results

The results of the screening tests are presented in Table 2.13-1. The temperatures reported in the table are the values for the exposed surface of the contaminant. The nitrogen trifluoride used in the tests had a minimum purity level of 98.2 weight percent nitrogen trifluoride and a maximum active fluoride content of 0.17 weight percent.

The significance of the data in Table 2.13-1 is that the candidate contaminants except for FC-75 are more reactive with gaseous NF_3 than with gaseous O_2 at comparable temperatures, but the reactions are not vigorous at the mild conditions existing in the test.

2.13.2.2 Adiabatic Compression Test Results

The results of the adiabatic compression tests are presented in Table 2.13-2. The threshold conditions at which no reaction occurs with either clean Nickel 200 or 347 stainless steel are incorporated into the table with the contaminant labeled as none and the values are 606 K (632 F) and 560 K (548 F) respectively.

The significant items to note from the data in Table 2.13-2 are as follows. The presence of fingerprints on Nickel 200 and 347 stainless steel lowered the threshold values 27 K (50 F) and 35 K (63 F) respectively. The presence of light weight machine oil (Walsco #985) on Nickel 200 and 347 stainless steel lowered the threshold values 17 K (32 F) and 19 K (34 F) respectively. The presence of FC-75 (3M) on Nickel 200 and 347 stainless steel lowered the threshold values 18 K (33 F) and 74 K (133 F) respectively. The presence of Brazing flux (Victor #3, general brazing) on Nickel 200 and 347 stainless steel lowered the threshold values 15 K (27 F) and 6 K (10 F) respectively. The presence of petroleum jelly (Vaseline) on the Nickel 200 and 347 stainless steel led to ignition of the petroleum jelly on the nickel with no apparent surface change while the threshold value of the 347 stainless steel was lowered 35 K (63 F), the same degree of lowering as observed in the presence of fingerprints. The data clearly demonstrate that the selected contaminants are detrimental to the compatibility of the metals with gaseous nitrogen trifluoride under adiabatic compression conditions.

TABLE 2.13-1

DATA INDICATIVE OF THE REACTIVITY OF CONTAMINANTS WITH NITROGEN TRIFLUORIDE AT ONE ATMOSPHERE PRESSURE AND IN COMPARISON WITH GASEOUS OXYGEN

Contaminant	Gas Present	Observations
Petroleum Jelly (Vaseline)	N_2	Slight changes in the slope of the heating curves at 364 K (195 F) and above. No apparent color change.
	O_2	Very slight changes in the slope of the heating curves at 323 K (121 F) and 325 K (143 F). No apparent color change.
	NF_3	Slight changes in heating curve slope as low as 314 K (105 F), color change occurred at temperatures as low as 380 K (225 F). By 478 K (400 F), its coloration was a dark-brown.
Brazing Flux (Victor #3 General Brazing)	O_2	No apparent reaction.
	NF_3	Color change from bright yellow to yellowish-brown at 372 K (210 F), slight changes in heating curves occurring above 383 K (230 F).
Fluorocarbon Oil (FC-75)	O_2	No changes in heating curves as the FC-75 gradually evaporated.
	NF_3	The same behavior as with O_2.
Light-Weight Machine Oil (Walco #985)	O_2	No changes in the heating curve below 483 K (410 F) but a slight color change occurred.
	NF_3	Slight change in slope of the heating curve at 414 K (285 F) accompanied by a color change.
Fingerprints on Silastic LS-53	O_2	No changes in slope of the heating curve below 478 K (400 F).
	NF_3	Slight changes in slope of the heating curves occurred at temperatures from 336 K (145 F) to 428 K (310 F).

TABLE 2.13-2

DATA INDICATIVE OF THE EFFECTS OF CONTAMINANTS ON METAL/NITROGEN TRIFLUORIDE COMPATIBILITY UNDER ADIABATIC COMPRESSION CONDITIONS IN GASEOUS NITROGEN TRIFLUORIDE

		Initial Conditions				Final Conditions				NF_3 Density		
		Temperature		Pressure		Temperature		Pressure				
Contaminant	Material	°K	°F	kN/m^2	psia	°K	°F	MN/m^2	psia	kg/m^3	lb/ft^3	Test Results
Fingerprints	Ni-200	289	61	33.8	4.9	606	632	8.38	1215	118	7.38	+, Slight surface change
Fingerprints	Ni-200	285	53	33.8	4.9	586	596	7.00	1015	102	6.37	+, Some surface change
Fingerprints	Ni-200	285	53	33.8	4.9	579	582	6.31	915	93.2	5.82	-
Fingerprints	Ni-200	285	53	33.8	4.9	570	567	5.62	815	84.3	5.26	-
None	Ni-200	290	62	33.8	4.9	606	632	8.38	1215	118	7.38	-
Fingerprints	347 SS	285	53	33.8	4.9	551	533	4.24	615	65.8	4.11	+, Slight surface change
Fingerprints	347 SS	285	53	33.8	4.9	539	510	3.55	515	56.4	3.52	+, Considerable surface change
Fingerprints	347 SS	285	53	33.8	4.9	525	485	2.86	415	46.6	2.91	-
None	347 SS	291	64	33.8	4.9	560	548	4.24	615	64.9	4.05	-
Machine Oil	Ni-200	286	55	33.8	4.9	603	625	8.38	1215	119	7.43	+, Slight surface change, oil ignited
Machine Oil	Ni-200	286	55	33.8	4.9	596	613	7.69	1115	110	6.89	+, Slight surface change
Machine Oil	Ni-200	286	55	33.8	4.9	589	600	7.00	1015	102	6.35	-
Machine Oil	347 SS	285	53	33.8	4.9	554	537	4.24	615	65.5	4.09	+, Some surface change
Machine Oil	347 SS	285	53	33.8	4.9	541	514	3.55	515	56.1	3.50	-
Machine Oil	347 SS	285	53	33.8	4.9	528	490	2.86	415	46.4	2.90	-
FC-75	Ni-200	285	53	33.8	4.9	600	621	8.38	1215	119	7.45	+, Considerable surface change
FC-75	Ni-200	286	55	33.8	4.9	594	610	7.69	1115	111	6.91	+, Very slight surface change
FC-75	Ni-200	286	55	33.8	4.9	588	599	7.00	1015	102	6.36	-
FC-75	347 SS	266	55	33.8	4.9	554	538	4.24	615	65.5	4.08	+, Some surface change
FC-75	347 SS	286	55	33.8	4.9	528	490	2.86	415	46.4	2.90	+, Slight surface change
FC-75	347 SS	286	55	33.8	4.9	509	457	2.17	315	36.5	2.78	+, Some surface change
FC-75	347 SS	286	55	33.8	4.9	486	415	1.48	215	26.1	1.63	-
Brazing Flux	Ni-200	283	50	33.8	4.9	598	617	8.38	1215	120	7.48	+, Some surface change
Brazing Flux	Ni-200	284	52	33.8	4.9	591	605	7.69	1115	111	6.94	-
Brazing Flux	Ni-200	284	52	33.8	4.9	586	595	7.00	1015	102	6.38	-
Brazing Flux	347 SS	286	55	33.8	4.9	574	573	5.62	815	83.8	5.23	+, Some surface change
Brazing Flux	347 SS	286	55	33.8	4.9	564	556	4.93	715	74.8	4.67	+, Slight surface change
Brazing Flux	347 SS	286	55	33.8	4.9	554	538	4.24	615	65.5	4.09	-
Petroleum Jelly	Ni-200	284	52	33.8	4.9	599	618	8.38	1215	120	7.47	+, Some surface change, contaminant ignited
Petroleum Jelly	Ni-200	284	52	33.8	4.9	593	608	7.69	1115	111	6.92	+, Contaminant ignited, surface unchanged
Petroleum Jelly	Ni-200	284	52	33.8	4.9	586	595	7.00	1015	42.3	6.38	+, Contaminant ignited, surface unchanged
Petroleum Jelly	347 SS	284	52	33.8	4.9	551	532	4.24	615	65.9	4.11	+, Slight surface change
Petroleum Jelly	347 SS	284	52	33.8	4.9	537	507	3.55	515	56.6	3.53	+, Slight surface change
Petroleum Jelly	347 SS	285	53	33.8	4.9	525	485	2.86	415	46.7	2.91	-

2.13, Effects of Contaminants on Metal Compatibility in Nitrogen Trifluoride (cont.)

2.13.2.3 Gaseous Flow Test Results

The data obtained from the tests in which the 316 ELC stainless steel was contaminated are presented in Table 2.13-3 and the data obtained from the tests in which Inconel 625 was contaminated are presented in Table 2.13-4. The threshold temperatures at which events occurred with uncontaminated samples are 911 K (1180 F) for the 316 ELC stainless steel and 950 K (1250 F) for the Inconel 625. The significant items to note from the data in Tables 2.13-3 and 2.13-4 are as follows.

First, the petroleum jelly does not affect the nitrogen trifluoride/metal compatibility under the test conditions but the contaminant and the nitrogen trifluoride can react explosively. Second, the nitrogen trifluoride/metal compatibility is not significantly altered by the presence of machine oil in the flow tests, but the nitrogen trifluoride/machine oil interactions do affect the flow conditions. Third, the fluorocarbon oil, FC-75, is apparently vaporized under the test conditions and no detrimental event occurs. Fourth, the presence of the brazing flux leads to thermal excursions in the flowing nitrogen trifluoride, the reaction threshold temperature with 316 ELC stainless steel was not lowered but the reaction threshold temperature for Inconel 625 was apparently lowered from 950 K (1250 F) to 875 K (1115 F). And fifth, the presence of fingerprints does not affect the nitrogen trifluoride/metal compatibility and there is probably insufficient contaminant present to indicate a nitrogen trifluoride/fingerprint interaction in the test configuration used. The data do demonstrate the need for adequate cleanliness precautions to avoid undesired upsets in the flowing NF_3 systems.

TABLE 2.13-3

DATA INDICATIVE OF THE EFFECTS OF VARIOUS CONTAMINANTS ON 316 ELC STAINLESS STEEL IN FLOWING GASEOUS NITROGEN TRIFLUORIDE

Material and Contaminant	Specimen Temperature °K	Specimen Temperature °F	Orifice Pressure Ratio	Upstream Pressure N/m^2	Upstream Pressure psia	Material Response	Test No.
316 ELC and Vaseline	466	~380	Sonic	1.35	196	Onset of rapidly increasing flow resistance	134
	594	~610	↓	1.56	226	Nondestructive explosion	134
	700	~800	↓	1.28	186	Flow resistance essentially back to normal	134
	1069	1465	↓	1.32	192	No abnormal behavior to max. test temp., orifice essentially unchanged	134
	450	~350	1.21	.81	117	Onset of rapidly increasing flow resistance	124
	578	~580	Sonic	.85	123	Max. flow resistance observed	124
	755	~900	1.31	.78	113	Flow resistance essentially back to normal	124
	1086	1495	1.43	.79	114	No abnormal behavior to max. test temp., orifice virtually unchanged	124
316 ELC and Machine Oil	466	~380	Sonic	1.34	194	Onset of abnormal increase in flow resistance	135
	589	~600	↓	1.52	220	Max. flow resistance and sudden relief	135
	666	~740	↓	1.28	185	Flow resistance essentially back to normal	135
	1083	1490	↓	1.34	194	No abnormal behavior to max. test temp., orifice virtually unchanged	135
	461	~370	1.21	.80	116	Onset of rapidly increasing flow resistance	125
	639	690	1.51	.83	120	Max. flow resistance observed	125
	805	990	1.35	.79	114	Flow resistance essentially back to normal	125
	1105	1530	1.49	.79	115	Major film buildup	125
	1139	1590	Sonic	.86	125	Minor film loss	125
	1155	1620	↓	.85	123	No failure to max. test temp., orifice virtually unchanged	125
316 ELC and Brazing Flux	644	700	Sonic	1.36	197	Onset of a sharp endotherm	137
	941	~1235	↓	1.37	199	Onset of a modest film buildup	137
	1086	1495	↓	1.39	201	No abnormal behavior to max. test temp., orifice virtually unchanged	137
	644	700	1.33	.78	113	Onset of a sharp endotherm	128
	933	~1220	1.34	.77	112	Onset of a minor film buildup	128
	1086	1495	1.55	.79	115	No abnormal behavior to max. test temp., orifice virtually unchanged	128
316 ELC and FC-75	1089	~1500	1.55	.77	111	No abnormal behavior to max. test temp., orifice virtually unchanged	126
	1089	~1500	Sonic	1.37	199	No abnormal behavior to max. test temp., orifice virtually unchanged	126
316 ELC and Fingerprints	1083	1490	1.61	.79	115	No abnormal behavior to max. test temp., orifice virtually unchanged	127

TABLE 2.13-4

DATA INDICATIVE OF THE EFFECTS OF VARIOUS CONTAMINANTS ON INCONEL 625 IN FLOWING GASEOUS NITROGEN TRIFLUORIDE

Material and Contaminant	Specimen Temperature °K	Specimen Temperature °F	Orifice Pressure Ratio	Upstream Pressure N/m^2	Upstream Pressure psia	Material Response	Test No.
Inconel 625 and Vaseline	466	~380	Sonic	1.33	193	Onset of rapidly increasing flow resistance	133
	594	~610		1.43	208	Max. flow resistance and sudden relief	133
	686	~775		1.29	187	Flow resistance back to normal	133
	969	1285		1.32	192	Sharp minor exotherm	133
	1089	1500		1.34	195	No abnormal behavior to max. test temp. orifice virtually unchanged	133
	466	~380	1.26	.74	107	Onset of a rapidly increasing flow resistance	119
	600	~620	Sonic	.79	115	Max. flow resistance and sudden relief	119
	766	~920	1.35	.72	104	Flow resistance back to normal	119
	1108	1535	1.52	.73	106	Minor endothermic film buildup and loss	119
	1144	1600	1.54	.73	106	Major endothermic film buildup	119
	1172	1650	Sonic	.77	111	Minor film loss and major reformation	119
	1214	1725	Sonic	.77	111	No abnormal behavior to max. test temp., orifice virtually unchanged	119
Inconel 625 and Machine Oil	464	375	Sonic	1.29	187	Onset at rapidly increasing flow resistance and a series of sharp exotherms	129
	622	660	Sonic	1.42	206	Max. flow resistance observed	129
	811	1000	Sonic	1.28	186	Flow resistance back to normal	129
	1097	1515	Sonic	1.34	194	No abnormal behavior to max. test temp., orifice virtually unchanged	129
	455	360	1.34	.74	108	Onset of rapidly increasing flow resistance	120
	680	765	Sonic	.79	115	Max. flow resistance observed	120
	950	1250	1.37	.72	105	Flow resistance back to normal	120
	1130	1575	1.45	.73	106	Major endothermic film buildup	120
	1155	1620	1.71	.74	108	Minor film loss and reformation	120
	1186	1675	1.70	.74	108	Minor film loss and reformation	120
	1208	1715	Sonic	.75	109	Minor film loss and reformation	120
	1244	1780	Sonic	.76	110	Minor film loss	120
	1275	1835	Sonic	.76	110	No failure to max. test temp., orifice virtually unchanged	120
Inconel 625 and Brazing Flux	639	690	Sonic	1.31	190	Minor film loss and sharp exotherm	132
	844	1060		1.32	192	Sharp minor exotherm	132
	875	1115		1.32	192	Sharp exotherm; minor film loss/reformation	132
	944	1240		1.34	194	Minor sharp exotherm	132
	1039	1410		1.35	196	Minor sharp exotherm and film loss	132
	1083	1490		1.34	195	Modest film loss, orifice virtually unchanged	132
	658	725	1.34	.74	108	Endo-/exothermic reactions and minor film loss	122
	1083	1490	1.40	.73	106	No unusual behavior to max. test temp., orifice virtually unchanged	122
Inconel 625 and FC-75	950	1250	Sonic	1.36	197	Minor film buildup	130
	1083	1490		1.37	199	Minor film loss, orifice virtually unchanged	130
	1086	1495	1.49	.73	106	No unusual behavior to max. test temp, orifice virtually unchanged	121
Inconel 625 and Fingerprints	1094	1510	Sonic	1.38	200	No unusual behavior to max. test temp., orifice virtually unchanged	131
	1083	1490	1.39	.79	115	No unusual behavior to max. test temp., orifice virtually unchanged	123

2.14 EFFECTS OF IMPURITIES IN NITROGEN TRIFLUORIDE COMPATBILITY WITH METALS

Two impurities which can readily occur in nitrogen trifluoride are hydrogen fluoride and water. The objective of this study was to determine the effect of these in nitrogen trifluoride on the chemical compatibility with selected metals.

2.14.1 The Effect of Hydrogen Fluoride in Nitrogen Trifluoride

Seven metals were selected for evaluation in the study: 2219 aluminum T-87, CRES 316 ELC, Inconel -625, Inconel 718, Nickel 200, C-1010 steel, and VM 250 maraging steel.

2.14.1.1 Apparatus and Procedures

The metal specimens were mounted as coupons on a rack and placed in a 5.1 cm (2 inches) diameter test containers as shown in Figures 2.1.1 and 2.1.2. The predetermined quantity of anhydrous hydrogen fluoride was transferred to the test container and then the nitrogen trifluoride was added. The test containers were then placed in the appropriate temperature bath.

2.14.1.2 Experimental Results

The periods of exposure were nominally 30, 90, and 230 days. One of the test containers, BHX, which was originally loaded with the equivalent of 1 weight percent hydrogen fluoride in nitrogen trifluoride at 344 K (160 F) and 3.45 MN/m^2 (500 psia) was removed from the temperature bath periodically to obtain a sample of the gas for chemical analysis. The container was recharged twice with the equivalent of 1 weight percent hydrogen fluoride. The sequence of events was as follows:

Day	Event
1	Container loaded to contain 1 w/o HF
18	Analysis indicates 0.0076 w/o HF
18	Additional 1 w/o HF added to container
19	Analysis indicates 0.24 w/o HF
19	Additional 1 w/o HF added to container
27	Analysis indicates 0.052 w/o HF, container removed from 344 K oven to ambient temperature environment
31	Analysis indicates 0.019 w/o HF

2.14, Effects of Impurities in Nitrogen Trifluoride Compatibility with Metals (cont.)

The data indicate that the hydrogen fluoride-metal reaction is relatively rapid at 344 K (160 F) with some of the metals present.

The data obtained for the various metals for the exposure periods are presented in Tables 2.14-1 through 2.14-7. The corrosion penetration rate values are given, but the values are misleading because the hydrogen fluoride is rapidly depleted in test container. For example, the data in Table 2.14-1 indicates that after 27 days exposure to the equivalent of 3 w/o hydrogen fluoride the average corrosion penetration rate of the 2219 aluminum is 33 pm/sec and that for 227 days of exposure the rate drops to 4.8 pm/sec. The associated average weight losses are 0.32 gm and 0.39 gm respectively. The data should be interpreted to indicate that the corrosion stops when the hydrogen fluoride is depleted. Further the aluminum surfaces were deeply pitted which invalidates a uniform corrosion penetration rate value.

The corrosion penetration rates for the liquid/vapor tests at 195 K (-78 C) indicates some enhancement of corrosion due to the presence of hydrogen fluoride in the nitrogen trifluoride. However, the rates are all well below 0.8 pm/sec (1 mpy) at initial hydrogen fluoride concentrations up to 1 weight percent. The initial hydrogen fluoride concentration is apparently depleted to 0.021 weight percent after 217 days. The corrosion rates of the maraging steel 250, Inconel 718 (STA), and aluminum 2219 T87 were enhanced to a greater degree by hydrogen fluoride contamination than the Nickel 200 and 316 ELC stainless steel. The Inconel 625 was apparently unaffected by the presence of the hydrogen fluoride under the test conditions.

The presence of hydrogen fluoride in nitrogen trifluoride at 344 K (160 F) causes severe corrosion of 2219 aluminum, maraging steel 250 and the 1010 steel, but only slight enhancement of the corrosion of 316 ELC, Nickel 200, and Inconels 718 and 625. It must be recognized however that the specimens were exposed in a ganged-manner and that the hydrogen fluoride was depleted rapidly via the corrosion of the less compatible metals. The corrosion rates reflect only qualitatively the relative corrosion resistance of the metals to the hydrogen fluoride-contaminated nitrogen trifluoride. The depletion of the hydrogen fluoride in the nitrogen trifluoride is demonstrated by the post-test analyses presented in Table 2.14-8.

The post-test condition of the metal specimens subjected to liquid/vapor exposure of nitrogen trifluoride contaminated with hydrogen fluoride is shown in Figure 2.14.1. The vapor exposed portion of the coupon has been exposed to very low hydrogen fluoride

TABLE 2.14-1

DATA INDICATIVE OF THE CORROSIVE EFFECT OF HYDROGEN FLUORIDE IN NITROGEN TRIFLUORIDE ON ALUMINUM 2219, T-87

Material -Specimen Type	Initial HF Content, Weight %	Time, Days	Exposure Conditions: Pressure Mn/m^2	Pressure psia	Temperature °K	Temperature °F	Specimen Surface Area, cm^2	Specimen Weight, gm: Initial	Final	Loss	Penetration Rate: pm/sec	mpy	Test No.	Observations
Al 2219, T-87 -Parent	0.28	28	Liquid/Vapor		195	-108	14.95	3.3702	3.3706	(0.0004)	0	0	AHX	Random dark spots
Al 2219, T-87 -Welded	0.28		Liquid/Vapor		195	-108	15.07	3.8982	3.8977	0.0005	0.051	0.063	AHX	Random dark spots
Al 2219, T-87 -Parent	1.0	91	Liquid/Vapor		195	-108	15.19	3.4940	3.4866	0.0074	0.23	0.29	AHY	Gray and black deposits
Al 2219, T-87 -Welded	1.0	91	Liquid/Vapor		195	-108	15.02	3.8324	3.8196	0.0128	0.41	0.50	AHY	Gray and black deposits
Al 2219, T-87 -Parent	1.0	217	Liquid/Vapor		195	-108	15.25	3.4164	3.4111	0.0053	0.069	0.086	AHZ	Drak gray stain with some pitting
Al 2219, T-87 -Welded	1.0	217	Liquid/Vapor		195	-108	15.19	3.9540	3.9473	0.0067	0.088	0.11	AHZ	Dark gray stain with some pitting
Al 2219, T-87 -Parent	1.0	34	3.45	500	294	70	14.95	3.3706	3.2221	0.1485	12.6	15.7	EHX	Two distinct coating layers: Rough, brown surface scale; Thick, flakey pinkish-white outer coating
Al 2219, T-87 -Welded	1.0	34	3.45	500	294	70	15.07	3.8977	3.8205	0.0772	6.5	8.1	EHX	Two distinct coating layers: Rough, brown surface scale; Thick, flakey pinkish-white outer coating
Al 2219, T-87 -Parent	0.1	25	3.45	500	344	160	14.95	1.6087	1.6050	0.0037	0.42	0.53	BH1X	Tarnished surface
Al 2219, T-87 -Parent	0.5	25	3.45	500	344	160	15.35	1.6436	1.5406	0.1030	11	14	BH5X	White film on surface
Al 2219, T-87 -Parent	1.0	90	3.45	500	344	160	15.20	3.3695	2.9757	0.3938	12.3	15.3	BHY	Badly corroded with severe pitting
Al 2219, T-87 -Welded	1.0	90	3.45	500	344	160	14.97	3.9043	3.5617	0.3426	10.9	13.5	BHY	Badly corroded with severe pitting
Al 2219, T-87 -Parent	3.0	227	3.45	500	344	160	15.22	3.4231	3.0373	0.3858	4.8	6.0	BHZ	Heavy white deposit with considerable pitting
Al 2219, T-87 -Welded	3.0	227	3.45	500	344	160	15.27	3.7643	3.3687	0.3956	4.9	6.1	BHZ	Heavy white deposit with considerable pitting
Al 2219, T-87 -Parent	3.0	27	3.45	500	344	160	15.14	3.4881	3.1862	0.3019	32	39	BHZ	General corrosion and pitting
Al 2219, T-87 -Welded	3.0	27	3.45	500	344	160	15.03	3.9300	3.5852	0.3348	35	44	BHX	General corrosion and pitting

TABLE 2.14-2

DATA INDICATIVE OF THE CORROSIVE EFFECT OF HYDROGEN FLUORIDE IN NITROGEN TRIFLUORIDE ON CRES 316 ELC STAINLESS STEEL

Material -Specimen Type	Initial HF Content, Weight %	Time, Days	Exposure Conditions: Pressure		Exposure Conditions: Temperature		Specimen Surface Area, cm^2	Specimen Weight, gm			Penetration Rate		Test No.	Observations
			Mn/m^2	psia	°K	°F		Initial	Final	Loss	pm/sec	mpy		
316 ELC -Parent	0.28	28	Liquid/Vapor		195	-108	14.45	2.0606	2.0592	0.0014	0.050	0.062	AHX	Tarnished appearance
316 ELC -Parent	1.0	91	Liquid/Vapor		195	-108	14.31	2.0591	2.0586	0.0005	0.006	0.007	AHY	Very light gray stain
316 ELC -Welded	1.0	91	Liquid/Vapor		195	-108	14.09	2.6809	2.6787	0.0022	0.025	0.031	AHY	Very light gray stain
316 ELC -Parent	1.0	217	Liquid/Vapor		195	-108	14.38	2.0805	2.0801	0.0004	0.002	0.002	AHZ	Very light purple stain
316 ELC -Welded	1.0	217	Liquid/Vapor		195	-108	14.04	3.0337	3.0323	0.0014	0.007	0.008	AHZ	Very light purple stain
316 ELC -Parent	1.0	34	3.45	500	294	70	14.45	2.0592	2.0524	0.0068	0.20	0.25	EHX	Random dark brown spots
316 ELC -Parent	0.1	25	3.45	500	344	160	14.27	2.0717	2.0716	0.0001	0.004	0.005	BH1X	Purplish-gold stains
316 ELC -Parent	0.5	25	3.45	500	344	160	14.33	2.0707	2.0615	0.0092	0.37	0.46	BH5X	Reddish-brown film on surface
316 ELC -Parent	1.0	90	3.45	500	344	160	14.29	2.0501	2.0440	0.0061	0.068	0.085	BHY	Covered with green-gray stain
316 ELC -Welded	1.0	90	3.45	500	344	160	14.15	2.8803	2.8757	0.0046	0.052	0.065	BHY	Covered with green-gray stain
316 ELC -Parent	3.0	27	3.45	500	344	160	14.31	2.0545	2.0325	0.0220	0.82	1.02	BHX	Brownish gray film
316 ELC -Parent	3.0	227	3.45	500	344	160	14.42	2.0440	2.0285	0.0155	0.069	0.085	BHZ	Yellow-green deposit
316 ELC -Welded	3.0	227	3.45	500	344	160	14.27	3.0538	3.0365	0.0173	0.077	0.096	BHZ	Yellow-green deposit

TABLE 2.14-3

DATA INDICATIVE OF THE CORROSIVE EFFECT OF HYDROGEN FLUORIDE IN NITROGEN TRIFLUORIDE ON INCONEL 625, ANNEALED

Material -Specimen Type	Initial HF Content, Weight %	Time, Days	Exposure Conditions: Pressure Mn/m^2	Exposure Conditions: Pressure psia	Exposure Conditions: Temperature °K	Exposure Conditions: Temperature °F	Specimen Surface Area, cm^2	Specimen Weight, gm: Initial	Specimen Weight, gm: Final	Specimen Weight, gm: Loss	Penetration Rate pm/sec	Penetration Rate mpy	Test No.	Observations
I-625 -Parent	0.28	28	Liquid/Vapor		195	-108	14.73	3.8518	3.8515	0.0003	0.010	0.013	AHX	No apparent reaction
I-625 -Parent	1.0	91	Liquid/Vapor		195	-108	14.98	3.9253	3.9248	0.0005	0.005	0.006	AHY	Light gray stain
I-625 -Welded	1.0	91	Liquid/Vapor		195	-108	14.87	4.3826	4.3821	0.0005	0.005	0.006	AHY	Light gray stain
I-625 -Parent	1.0	217	Liquid/Vapor		195	-108	14.93	3.9544	3.9541	0.0003	0.001	0.002	AHZ	Very slight purple color with small yellow spots
I-625 -Welded	1.0	217	Liquid/Vapor		195	-108	14.77	4.4523	4.4519	0.0004	0.002	0.002	AHZ	Very slight purple color with small yellow spots
I-625 -Parent	1.0	34	3.45	500	294	70	14.73	3.8515	3.8518	(0.0003)	0	0	EHX	Thin pink film with random green and blue spots
I-625 -Parent	0.1	25	3.45	500	344	160	14.86	3.9188	3.9182	0.0006	0.022	0.027	BH1X	No apparent reaction
I-625 -Parent	0.5	25	3.45	500	344	160	15.03	3.9762	3.9692	0.0070	0.25	0.32	BH5X	Light gray-green film
I-625 -Parent	1.0	90	3.45	500	344	160	14.85	3.8722	3.8692	0.0030	0.031	0.038	BHY	Covered with purple, green, and gray stains
I-625 -Welded	1.0	90	3.45	500	344	160	14.85	4.4041	4.4001	0.0040	0.041	0.051	BHY	Covered with purple, green, and gray stains
I-625 -Parent	3.0	27	3.45	500	344	160	14.85	3.9039	3.8858	0.0181	0.62	0.76	BHX	Yellow-green film
I-625 -Parent	3.0	227	3.45	500	344	160	14.68	3.8725	3.8539	0.0186	0.077	0.095	BHZ	Heavy yellow-green deposit
I-625 -Welded	3.0	227	3.45	500	344	160	14.83	4.5767	4.5570	0.0197	0.080	0.10	BHZ	Heavy yellow-green deposit

TABLE 2.14-4

DATA INDICATIVE OF THE CORROSIVE EFFECT OF HYDROGEN FLUORIDE IN NITROGEN TRIFLUORIDE ON INCONEL 718, STA

Material -Specimen Type	Initial HF Content, Weight %	Time, Days	Exposure Conditions: Pressure Mn/m^2	Pressure psia	Temperature °K	Temperature °F	Specimen Surface Area, cm^2	Specimen Weight, gm: Initial	Final	Loss	Penetration Rate: pm/sec	mpy	Test No.	Observations
I-718 -Parent	0.28	28	Liquid/Vapor		195	-108	14.61	3.6551	3.6530	0.0021	0.072	0.089	AHX	Slight dark stain
I-718 -Parent	0.28	28	Liquid/Vapor		195	-108	14.38	3.5959	3.5924	0.0035	0.12	0.15	AHX	Slight dark stain
I-718 -Parent	1.0	91	Liquid/Vapor		195	-108	14.54	3.6200	3.6180	0.0020	0.021	0.027	AHY	No apparent reaction
I-718 -Parent	1.0	217	Liquid/Vapor		195	-108	14.44	3.5306	3.5275	0.0031	0.014	0.017	AHZ	Black stain with some white spots and some pitting
I-718 -Welded	1.0	217	Liquid/Vapor		195	-108	14.28	3.6580	3.6381	0.0199	0.091	0.11	AHZ	Black stain with some white spots and some pitting
I-718 -Parent	1.0	34	3.45	500	294	70	14.38	3.5924	3.5920	0.0004	0.01	0.01	EHX	No apparent reaction
I-718 -Parent	0.1	25	3.45	500	344	160	14.23	1.4145	1.4134	0.0011	0.043	0.054	BH1X	No apparent reaction
I-718 -Parent	0.5	25	3.45	500	344	160	14.18	1.4062	1.4034	0.0028	0.11	0.14	BH5X	No apparent reaction
I-718 -Parent	1.0	90	3.45	500	344	160	14.67	3.6557	3.6539	0.0018	0.019	0.024	BHY	Covered with green and dark gray stain
I-718 -Welded	1.0	90	3.45	500	344	160	14.47	3.8757	3.8731	0.0026	0.028	0.035	BHY	Covered with green and dark gray stain
I-718 -Parent	3.0	227	3.45	500	344	160	14.49	3.6185	3.6014	0.0170	0.073	0.091	BHZ	Yellow-green deposit

TABLE 2.14-5

DATA INDICATIVE OF THE CORROSIVE EFFECT OF HYDROGEN FLUORIDE IN NITROGEN TRIFLUORIDE ON NICKEL 200, ANNEALED

Material -Specimen Type	Initial HF Content, Weight %	Time, Days	Exposure Conditions: Pressure Mn/m^2	Exposure Conditions: Pressure psia	Exposure Conditions: Temperature °K	Exposure Conditions: Temperature °F	Specimen Surface Area, cm^2	Specimen Weight, gm: Initial	Specimen Weight, gm: Final	Specimen Weight, gm: Loss	Penetration Rate: pm/sec	Penetration Rate: mpy	Test No.	Observations
Ni 200 -Parent	0.28	28	Liquid/Vapor		195	-108	14.19	1.5306	1.5303	0.0003	0.010	0.012	AHX	Random stains
Ni 200 -Welded	0.28	28	Liquid/Vapor		195	-108	14.19	2.1351	2.1346	0.0005	0.016	0.020	AHX	Random stains
Ni 200 -Parent	1.0	91	Liquid/Vapor		195	-108	14.15	1.5307	1.5298	0.0009	0.009	0.011	AHY	Purple stains
Ni 200 -Welded	1.0	91	Liquid/Vapor		195	-108	14.36	2.2170	2.2159	0.0011	0.011	0.014	AHY	Purple stains
Ni 200 -Parent	1.0	217	Liquid/Vapor		195	-108	14.28	1.-542	1.5532	0.0010	0.0042	0.0052	AHZ	Light purple stains most evident in liquid phase accompanied by slight pitting
NI 200 -Welded	1.0	217	Liquid/Vapor		195	-108	14.21	2.2723	2.2710	0.0013	0.0055	0.0068	AHZ	Light purple stains most evident in liquid phase
Ni 200 -Parent	1.0	34	3.45	500	294	70	14.19	1.5303	1.5291	0.0012	0.032	0.040	EHX	Random purple stains
Ni 200 -Welded	1.0	34	3.45	500	294	70	14.19	2.1346	2.1341	0.0005	0.01	0.02	EHX	Random purple stains
Ni 200 -Parent	0.1	25	3.45	500	344	160	14.19	1.5479	1.5478	0.0001	0.004	0.005	BH1X	Some stains
Ni 200 -Parent	0.5	25	3.45	500	344	160	14.21	1.4466	1.4430	0.0036	0.13	0.16	BH5X	Gray-green coating
Ni 200 -Parent	1.0	90	3.45	500	344	160	14.28	1.5456	1.5367	0.0089	0.090	0.112	BHY	Gray coating
Ni 200 -Welded	1.0	90	3.45	500	344	160	14.24	2.3430	2.3318	0.0112	0.114	0.141	BHY	Gray coating
Ni 200 -Parent	3.0	27	3.45	500	344	160	14.14	1.5276	1.4999	0.0286	0.97	1.21	BHX	Yellow-green film with black spots
Ni 200 -Welded	3.0	27	3.45	500	344	160	14.28	2.2095	2.1856	0.0239	0.80	1.00	BHX	Yellow-green film with black spots
Ni 200 -Parent	3.0	227	3.45	500	344	160	14.17	1.5394	1.5173	0.0221	0.089	0.11	BHZ	Green deposit with some pitting
Ni 200 -Welded	3.0	227	3.45	500	344	160	14.12	2.2937	2.2629	0.0308	0.12	0.16	BHZ	Green deposit with some pitting

TABLE 2.14-6

DATA INDICATIVE OF THE CORROSIVE EFFECT OF HYDROGEN FLUORIDE IN NITROGEN TRIFLUORIDE ON VM 250 MARAGING STEEL

Material -Specimen Type	Initial HF Content, Weight %	Time, Days	Exposure Conditions: Pressure Mn/m^2	Exposure Conditions: Pressure psia	Exposure Conditions: Temperature °K	Exposure Conditions: Temperature °F	Specimen Surface Area, cm^2	Specimen Weight, gm: Initial	Specimen Weight, gm: Final	Specimen Weight, gm: Loss	Penetration Rate pm/sec	Penetration Rate mpy	Test No.	Observations
VM 250 -Parent	0.28	28	Liquid/Vapor		195	-108	15.79	8.7700	8.7654	0.0046	0.153	0.190	AHX	Dark stain
VM 250 -Welded	0.28	28	Liquid/Vapor		195	-108	16.15	11.4800	11.4744	0.0056	0.182	0.226	AHX	Dark stain
VM 250 -Parent	1.0	91	Liquid/Vapor		195	-108	16.16	11.1109	11.1033	0.0076	0.077	0.095	AHY	Gray and black stain
VM 250 -Welded	1.0	91	Liquid/Vapor		195	-108	15.81	10.7536	10.7450	0.0086	0.089	0.110	AHY	Gray and black stain
VM 250 -Parent	1.0	217	Liquid/Vapor		195	-108	16.33	10.6157	10.6046	0.0111	0.046	0.057	AHZ	Gray stain most intense in liquid phase
VM 250 -Welded	1.0	217	Liquid/Vapor		195	-108	16.02	10.7364	10.7246	0.0117	0.049	0.061	AHZ	Gray stain most intense in liquid phase
VM 250 -Parent	1.0	34	3.45	500	294	70	15.79	8.7654	8.6990	0.0664	1.82	2.26	EHX	Three distinct coating layers: Blue surface coating; White middle layer; Rough, brown outer scale
VM 250 -Welded	1.0	34	3.45	500	294	70	16.15	11.4744	11.2978	0.1766	4.73	5.87	EHX	Three distinct coating layers; Blue surface coating; White middle layer; Rough, brown outer scale
VM 250 -Parent	0.1	25	3.45	500	344	160	16.52	10.9729	10.9594	0.0135	0.48	0.60	BH1X	Gray-black coating over surface
VM 250 -Parent	0.5	25	3.45	500	344	160	16.14	10.8830	10.8403	0.0427	1.6	1.9	BH5X	Heavy, dark gray-black deposit on surface
VM 250 -Parent	1.0	90	3.45	500	344	160	16.26	11.2727	10.7858	0.4869	4.89	6.08	BHY	Layer of purplish black film
VM 250 -Welded	1.0	90	3.45	500	344	160	15.85	10.4085	9.9990	0.4095	4.22	5.24	BHY	Layer of purplish black film
VM 250 -Parent	3.0	27	3.45	500	344	160	16.01	10.4789	10.3620	0.1169	3.97	4.93	BHX	Light purple-brown-black coating
VM 250 -Welded	3.0	27	3.45	500	344	160	16.12	10.9435	10.8300	0.1135	3.83	4.76	BHX	Light purple-brown-black coating
VM 250 -Parent	3.0	227	3.45	500	344	160	16.31	11.1313	9.3083	1.8230	7.24	8.99	BHZ	Very heavy green-black deposit
VM 250 -Welded	3.0	227	3.45	500	344	160	15.98	10.7842	9.1617	1.6225	6.58	8.16	BHZ	Very heavy green-black deposit

TABLE 2.14-7

DATA INDICATIVE OF THE CORROSIVE EFFECT OF HYDROGEN FLUORIDE IN NITROGEN TRIFLUORIDE ON C 1010 STEEL

Material -Specimen Type	Initial HF Content, Weight %	Time, Days	Exposure Conditions: Pressure Mn/m^2	Exposure Conditions: Pressure psia	Exposure Conditions: Temperature °K	Exposure Conditions: Temperature °F	Specimen Surface Area, cm^2	Specimen Weight, gm: Initial	Specimen Weight, gm: Final	Specimen Weight, gm: Loss	Penetration Rate: pm/sec	Penetration Rate: mpy	Test No.	Observations
C 1010 -Parent	0.28	28	Liquid/Vapor		195	-108	14.06	1.3302	1.3301	0.0001	0.004	0.005	AHX	Stained
C 1010 -Parent	1.0	91	Liquid/Vapor		195	-108	14.10	1.3228	1.3205	0.0023	0.027	0.033	AHY	Purple stain
C 1010 -Parent	1.0	217	Liquid/Vapor		195	-108	14.22	1.3420	1.3397	0.0023	0.011	0.014	AHZ	Gray-purple stain showing increased intensity together with pitting in the liquid phase
C 1010 -Welded	1.0	217	Liquid/Vapor		195	-108	13.99	1.6728	1.6692	0.0036	0.017	0.022	AHZ	Gray-purple stain showing increased intensity together with pitting in the liquid phase
C 1010 -Parent	1.0	34	3.45	500	294	70	14.06	1.3301	1.1989	0.1312	4.04	5.02	EHX	Two distinct coating layers: Rough, brown surface scale Chalky, blue-white outer coating
C 1010 -Parent	0.1	25	3.45	500	344	160	13.97	1.3302	1.3298	0.0004	0.017	0.021	BH1X	Purple-gold stains
C 1010 -Welded	0.5	25	3.45	500	344	160	14.07	1.6259	1.6213	0.0046	0.19	0.24	BH5X	Red-brown deposit on surface
C 1010 -Parent	1.0	90	3.45	500	344	160	14.11	1.3389	1.1268	0.2121	2.46	3.05	BHY	Layer of purplish-black film
C 1010 -Welded	1.0	90	3.45	500	344	160	14.03	1.7944	1.6023	0.1921	2.24	2.78	BHY	Layer of purplish-black film
C 1010 -Parent	3.0	27	3.45	500	344	160	14.11	1.3389	1.2735	0.0654	2.54	3.16	BHX	Layer of purplish-black film
C 1010 -Parent	3.0	227	3.45	500	344	160	14.08	1.3277	1.0461	0.2816	1.30	1.61	BHZ	Gray-green deposit

TABLE 2.14-8

CHEMICAL COMPOSITION OF NITROGEN TRIFLUORIDE RECOVERED FROM STATIC TESTS WITH HYDROGEN FLUORIDE

Test No.	Initial HF Content, Weight %	Exposure Conditions: Time, Days	Pressure MN/m^2	Pressure psia	Temperature °K	Temperature °F	Compsotion, Weight Percent: NF_3	Active Fluorides as HF	N_2	O_2/CO	CF_4	CO_2	N_2O	H_2	Original Cylinder No. of NF_3 Used in Test
AHX	0.28	28	Liquid/Vapor		195	-108	99.65	0.021	0	0.25	0.0064	0.0062	0.072	<0.0012	H81136
AHY	1.0	91	Liquid/Vapor		195	-108	99.61	0.0071	0	0.31	0.014	0	0.052	0.0021	P178684
AHZ	1.0	217	Liquid/Vapor		195	-108	99.42	0.021	0	0.47	0.012	0	0.082	<0.0012	H81136
EHX	1.0	34	3.45	500	294	70	99.60	0.26	0.031	0.015	0.016	0.010	0.062	0.013	P178684
BH1X	0.1	25	3.45	500	344	160	99.04	0.0023	0.068	0.18	0.65	0.013	0.043	<0.0012	Mixed Batch
BH5X	0.5	25	3.45	500	344	160	99.13	<0.0001	0.15	Trace	0.66	0.0095	0.036	0.0097	Mixed Batch
BHY	1.0	90	3.45	500	344	160	99.43	0.18	0.26	0.0015	0.016	0.0076	0.10	0.0021	P178684
BHZ	3.0	227	3.45	500	344	160	98.30	0.033	1.12	0	0.0096	0.0075	0.50	0.0096	H81136
BHX	3.0	27	3.45	500	344	160	99.78	0.019	0.080	0	0.0086	0.0041	0.085	0.016	H81136

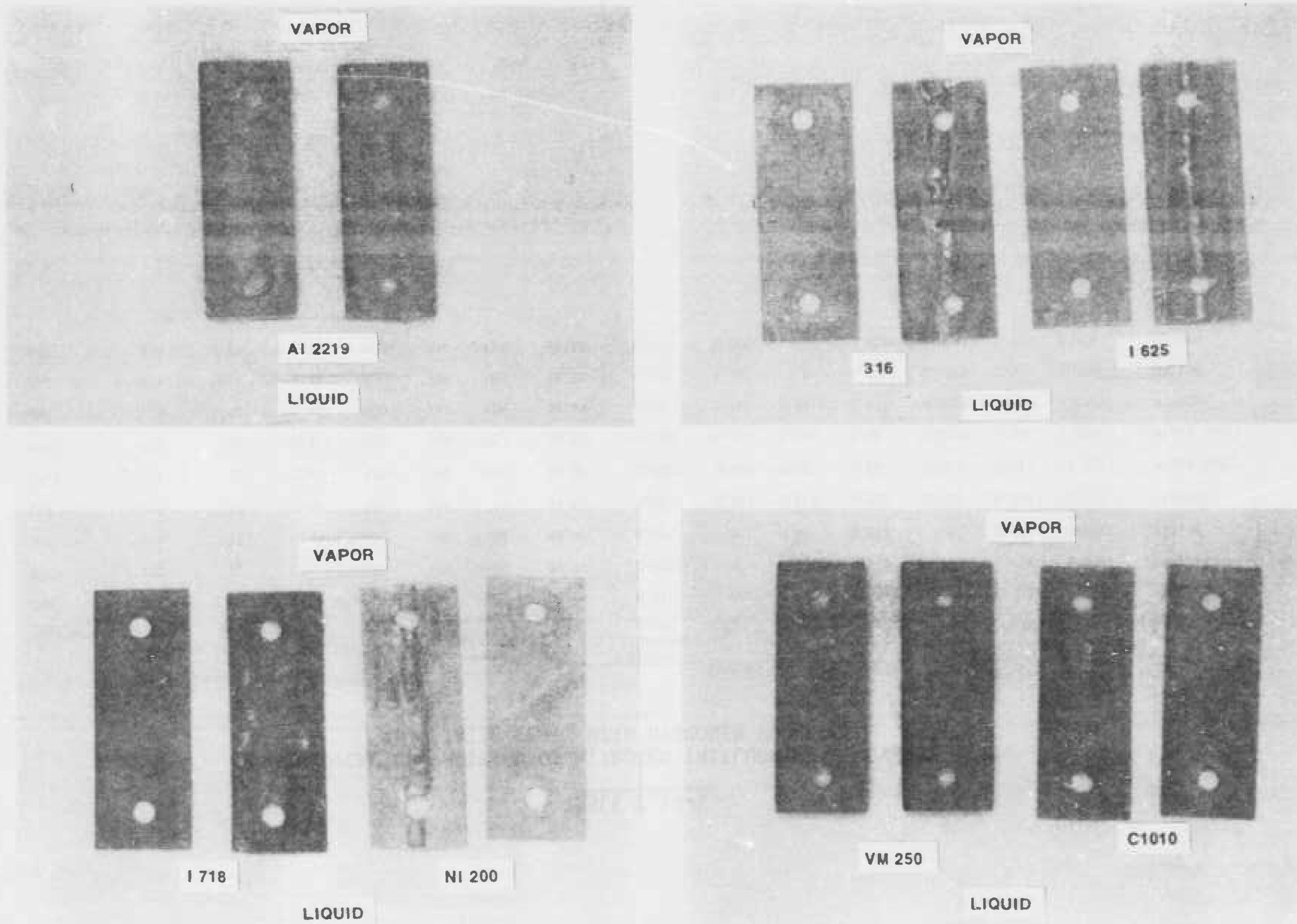


Figure 2.14.1. Metal Specimens After 217 Days Static Exposure to 1 Weight Percent Hydrogen Fluoride in Liquid/Vapor Nitrogen Trifluoride at 195°K (-108°F)

concentrations because the majority of the hydrogen fluoride is present in the liquid phase.

The corrosivity of hydrogen fluoride-contaminated nitrogen trifluoride at 344 K (160 F) is vividly demonstrated in Figure 2.14.2. The metal specimens subjected to the same conditions for 227 days are shown in Figure 2.14.3. By comparison of the photographs one can observe that the degree of attack is very similar between 27 days and 227 days. This further substantiates that the hydrogen fluoride is depleted significantly during the initial period of exposure.

The implication of the preceding findings is that the hydrogen fluoride concentration in nitrogen trifluoride systems should be minimized as much as possible in order to avoid significant corrosion of metals. In addition some hydrogen (see Table 2.14-8) is generated by the corrosion reactions which in turn can form a hazardous gas mixture.

2.14.2 The Effect of Water in Nitrogen Trifluoride

The effects of water on nitrogen trifluoride compatibility with metals was briefly investigated in two ways, (1) excess liquid water in the presence of nitrogen trifluoride and (2) low levels of water vapor in gaseous nitrogen trifluoride.

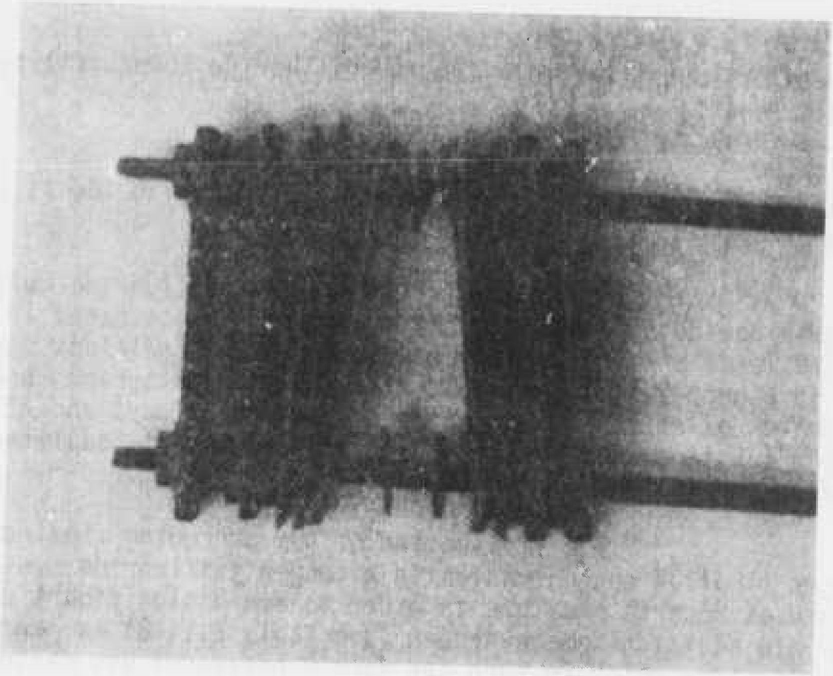
2.14.2.1 Apparatus and Procedures

The apparatus and procedures were similar to those described in Section 2.14.1.1.

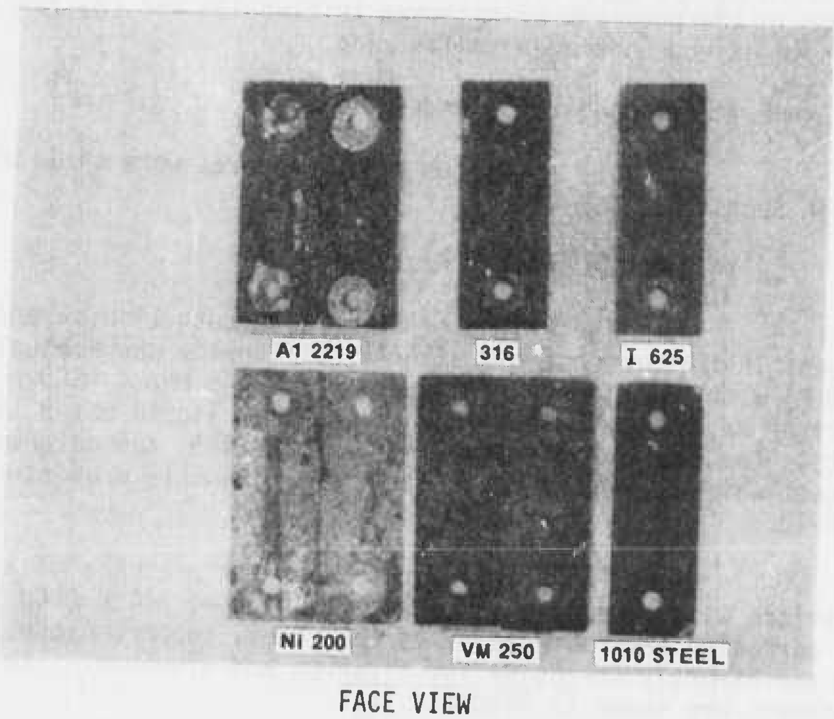
2.14.2.2 Experiment Results

The effect of water with both liquid- and vapor-phase present in the presence of nitrogen trifluoride was evaluated at 344 K (160 F) with the nitrogen trifluoride at 3.45 MN/m^2 (500 psia). The test container was approximately half-filled with liquid water to accentuate the effects. The data are presented in Table 2.14-9; the data with only water present which served as the control experiment is presented in Table 2.14-10.

The data indicate that the presence of a liquid water interface with gaseous nitrogen trifluoride at 344 K (160 F) is an extremely corrosive environment for carbon steel, copper, nickel, and Monel 400 and strongly corrosive for titanium and aluminum. The post-test metal specimens are shown in Figure 2.14-4.



SIDE VIEW



FACE VIEW

Figure 2.14.2. Photographs of Specimen Removed From Container BHX After Exposure to 3% HF in NF_3 for 27 Days

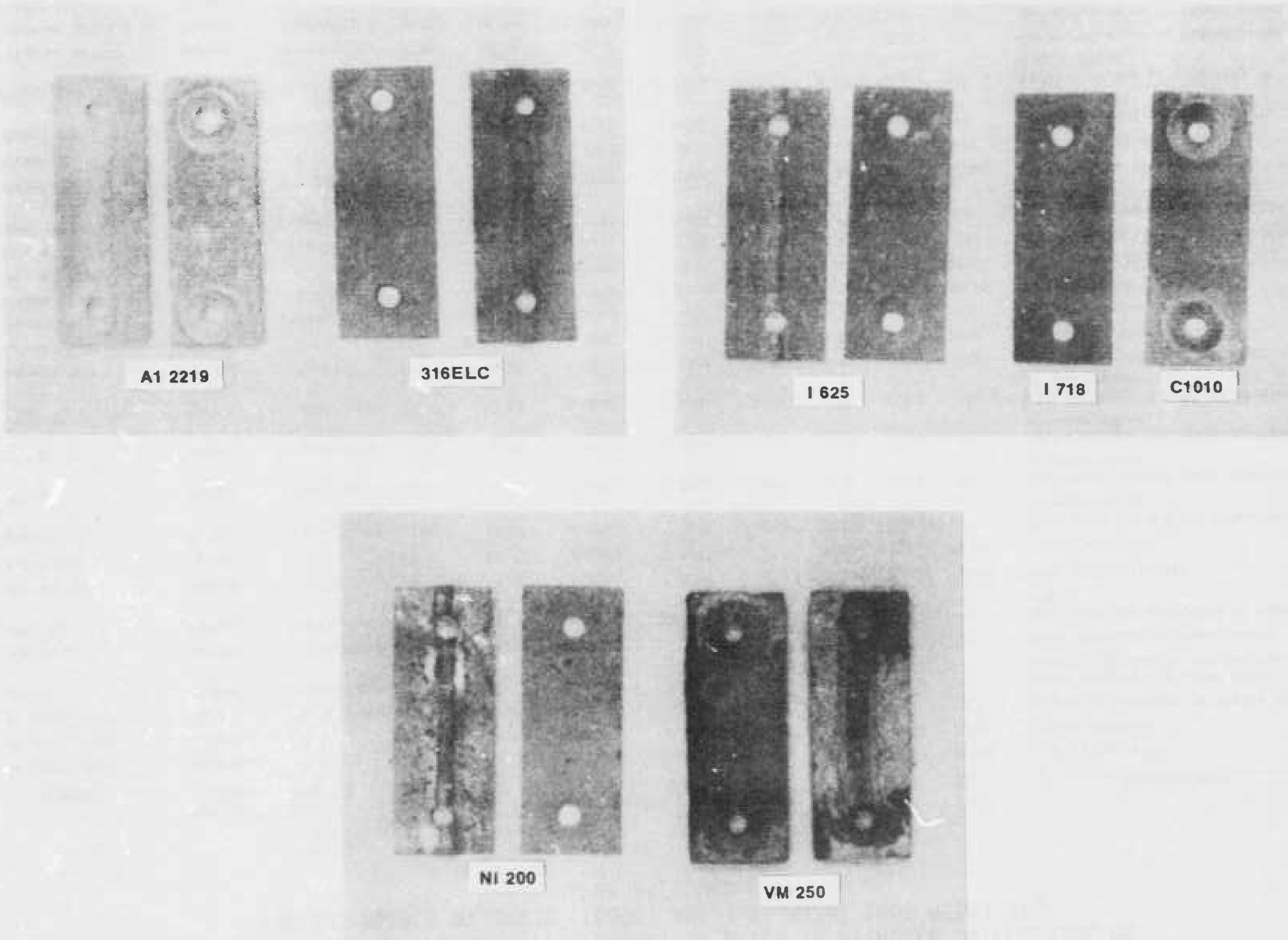


Figure 2.14.3. Metal Specimens After 227 Days Static Exposure to 3 Weight Percent Hydrogen Fluoride in Nitrogen Trifluoride at 3.45 MN/m^2 (500 psia) and 344 °K (160°F)

TABLE 2.14-9

DATA INDICATIVE OF THE CORROSIVE EFFECT OF WATER IN NITROGEN TRIFLUORIDE ON VARIOUS METALS AT 344°K (160°F) AND 3.45 MN/m² (500 PSIA) NF_3 VAPOR PRESSURE

Material	Specimen Type	Initial H_2O Content, Weight %	Exposure Time, Days	Specimen Surface Area, cm^2	Specimen Weight, gm Initial	Final	Loss	Penetration Rate pm/sec	mpy	Test No.	Observations
Al 2219, T-87	Welded	0.032	29	15.00	1.9037	1.8914	0.0123	1.21	1.50	BVLWX	Light gray stain
Al 2219, T-87	Welded	0.1	25	14.99	2.0095	2.0057	0.0038	0.43	0.54	BW1X	Slight tarnish
Al 6061, T-6	Parent	Liquid/Vapor	33	14.42	0.4841	0.4638	0.0203	1.82	2.26	20	Corrosion greatest in liquid phase
304 SS	Parent	Liquid/Vapor	33	14.06	1.3783	1.3572	0.0211	0.66	0.82	21	Major deposits in vapor phase, greatest attack in liquid phase
304 SS	Welded	Liquid/Vapor	33	14.06	1.7592	1.7319	0.0273	0.85	1.1	21	Major deposits in vapor phase
304L SS	Parent	Liquid/Vapor	33(9)	13.93	0.8476	0.8395	0.0081	0.25	0.32	19	Rust deposits and stain in vapor phase
316 ELC SS	Welded	0.032	29	14.31	2.8062	2.8043	0.0019	0.066	0.082	BVLWX	Very slight tarnish
316 ELC SS	Welded	0.1	25	14.34	2.8689	2.8678	0.0011	0.044	0.055	BW1X	Slight tarnish
316 ELC SS	Parent	Liquid/Vapor	33(9)	14.21	2.0655	2.0587	0.0068	0.21	0.26	19	Rust deposits and stain heaviest in vapor phase
321 SS	Parent	Liquid/Vapor	33(9)	14.23	2.0759	2.0686	0.0073	0.22	0.28	19	Rust deposits and stain heaviest in vapor phase
347 SS	Parent	Liquid/Vapor	33(9)	14.06	1.4236	1.4235	0.0001	0.03	0.04	19	Rust deposits and stain heaviest in vapor phase
17-4 PH, H-1025	Parent	Liquid/Vapor	33(9)	15.66	8.3580	8.3502	0.0078	0.23	0.28	19	Rust deposits and stains heaviest in vapor phase
Inconel 625	Welded	0.032	29	14.84	4.4502	4.4429	0.0073	0.23	0.29	BVLWX	Very light yellow-green deposit
Inconel 625	Welded	0.1	25	14.84	4.5063	4.4957	0.0106	0.39	0.49	BW1X	Gray-green film
Inconel 625	Parent	Liquid/Vapor	33(9)	14.57	3.9639	3.9639	0	0	0	19	Some stain and rust spots
Inconel 718	Welded	0.032	29	14.19	2.2217	2.1959	0.0258	0.88	1.1	BVLWX	Black deposit
Inconel 718	Welded	0.1	25	14.18	1.9278	1.9118	0.0160	0.63	0.79	BW1X	Black deposit
Inconel 718	Parent	Liquid/Vapor	33(9)	14.06	1.4377	1.4136	0.0241	0.73	0.90	19	Considerable stain and rust deposits
Monel 400	Parent	Liquid/Vapor	33(9)	14.21	2.2795	1.9905	0.2890	8.11	10.1	19	Badly corroded particularly in liquid phase
Nickel 200	Welded	0.032	29	14.15	2.1214	2.1084	0.0130	0.41	0.51	BVLWX	Light yellow-green deposit
Nickel 200	Welded	0.1	25	14.26	2.3528	2.3417	0.0111	0.40	0.50	BW1X	Gray-green film
Nickel 200	Parent	Liquid/Vapor	33(9)	14.05	1.5372	1.1996	0.3376	9.48	11.8	19	Badly corroded particularly in liquid phase
Nickel 270	Parent	Liquid/Vapor	33(9)	17.02	16.4652	16.1115	0.3537	8.20	10.2	19	Badly corroded particularly in liquid phase
Titanium 6Al-4V	Parent	Liquid/Vapor	33(9)	14.72	2.4077	2.3557	0.0520	2.8	3.5	19	Corrosion worse in liquid phase
Titanium 5Al-2.5 Sn	Parent	Liquid/Vapor	33(9)	14.68	2.3697	2.3296	0.0401	2.1	2.6	19	Corrosion worse in liquid phase
C-1010 Steel	Parent	0.1	25	14.10	1.3244	1.3189	0.0055	0.23	0.28	BW1X	Red-brown film

TABLE 2.14-9 (cont.)

Material	Specimen Type	Initial H_2O Content, Weight %	Exposure Time, Days	Specimen Surface Area, cm^2	Specimen Weight, gm			Penetration Rate		Test No.	Observations
					Initial	Final	Loss	pm/sec	mpy		
C-1010 Steel	Parent	Liquid/Vapor	33(9)	14.05	1.3045	0.6422	0.6623	21.2	26.3	19	Very little material left in liquid phase
Copper, OFHC	Parent	Liquid/Vapor	33(9)	14.06	1.5707	1.1541	0.4166	11.5	14.3	19	Corroded through at L/V interface
VM 250 Steel	Welded	0.032	29	16.17	11.6266	11.6148	0.0118	0.38	0.47	BVLWX	Heavy rusty brown deposit
VM 250 Steel	Welded	0.1	25	16.34	11.7236	11.7190	0.0046	0.17	0.21	BWIX	Brown-gray film

TABLE 2.14-10

DATA INDICATIVE OF THE COMPATIBILITY OF LIQUID/VAPOR WATER WITH VARIOUS METALS AT 344°K (160°F)

Material	Specimen Type	Exposure Time, Days	Specimen Surface Area, cm^2	Specimen Weight, gm			Penetration Rate		Observations
				Initial	Final	Loss	pm/sec	mpy	
Al 6061, T-6	Parent	33	14.42	0.4810	0.4733	0.0077	0.69	0.86	Coated with corrosion products
304 SS	Parent	33	14.06	1.3768	1.3768	0	0	0	No apparent reaction
304 SS	Welded	33	14.06	2.0704	2.0703	0.0001	0.0031	0.0039	No apparent reaction
304 L SS	Parent	35	13.93	0.8270	0.8270	0	0	0	No apparent reaction
316 ELC SS	Parent	35	14.21	2.0641	2.0640	0.0001	0.0029	0.0036	Some stain in vapor phase
321 SS	Parent	35	14.23	2.0568	2.0567	0.0001	0.0029	0.0036	Some stain
347 SS	Parent	35	14.06	1.4236	1.4235	0.0001	0.0029	0.0037	Slight stain in vapor phase
17-4 PH, H-1025	Parent	35	15.66	8.4658	8.4657	0.0001	0.0027	0.0034	Slight stain in vapor phase
Inconel 625	Parent	35	14.57	3.9639	3.9639	0	0	0	Slight stain in vapor phase
Inconel 718	Parent	35	14.06	1.4774	1.4774	0	0	0	Slight stain in vapor phase
Monel 400	Parent	35	14.21	2.2348	2.2347	0.0001	0.0026	0.0033	Some stain
Nickel 200	Parent	35	14.05	1.5247	1.5245	0.0002	0.0053	0.0066	Stain at L/V interface
Nickel 270	Parent	35	17.02	16.4197	16.4094	0.0103	0.23	0.28	Stain in liquid phase
Ti 6Al-4V	Parent	35	14.72	2.3977	2.3946	0.0033	0.17	0.21	Slight stain in liquid phase
Ti 5Al-2.5 Sn	Parent	35	14.68	2.3657	2.3658	(0.0001)	0	0	Stain in liquid phase
C-1010 Steel	Parent	35	14.05	1.3102	1.3028	0.0074	0.22	0.28	Black deposits on sample
Copper, OFHC	Parent	35	14.06	1.5521	1.5519	0.0002	0.0052	0.0065	Considerable tarnish in vapor phase

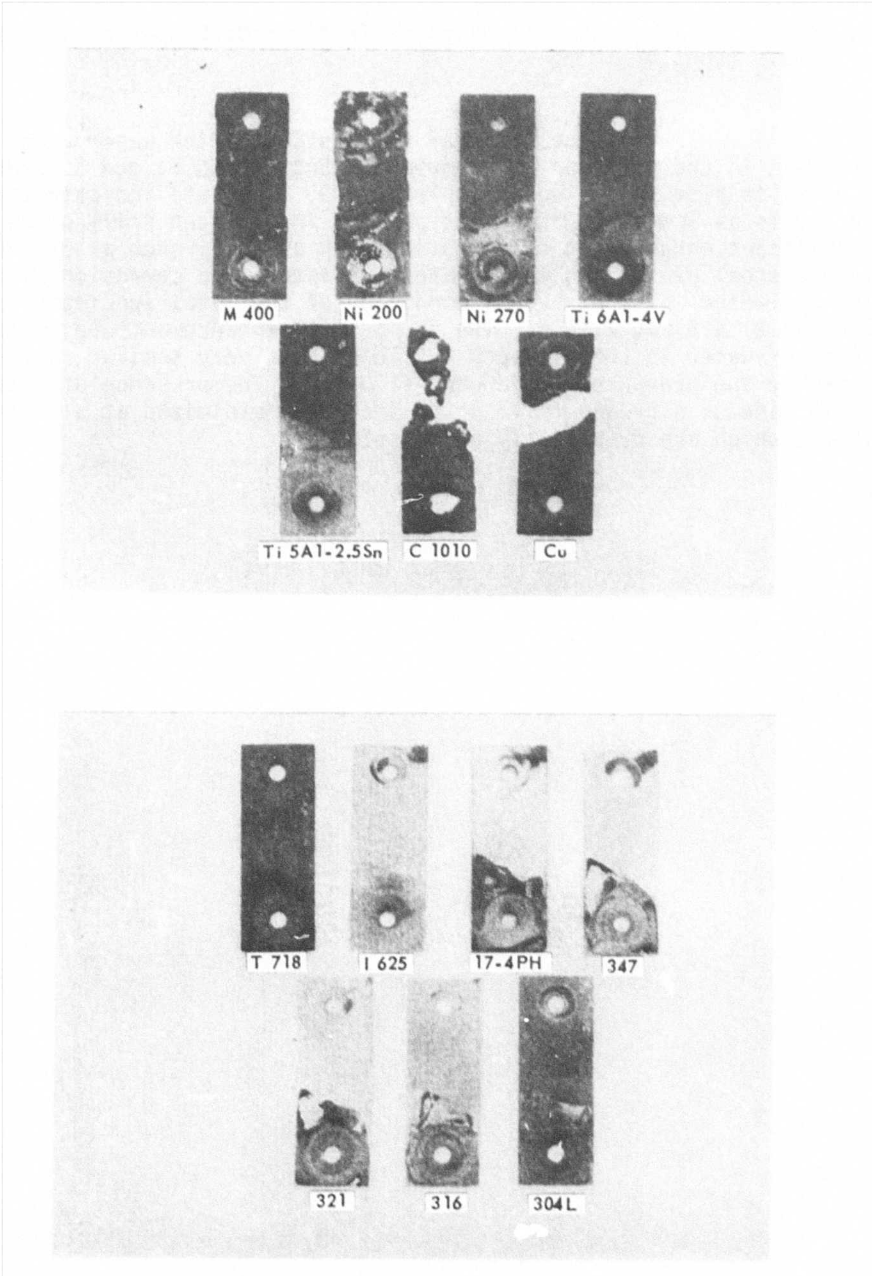


Figure 2.14.4. Metal Specimens After 33 Days Static Exposure to Liquid/Vapor Water With Nitrogen Trifluoride Present at 3.45 MN/m^2 (500 psia) and 344 °K (160°F). The Upper Portion of the Specimens in the Photographs Were Immersed in Liquid Water

2.14, Effects of Impurities in Nitrogen Trifluoride Compatibility with Metals (cont.)

The data for the tests in which water vapor only was present in the nitrogen trifluoride at 344 K (160 F) and 3.45 MN/m^2 (500 psia) is also presented in Table 2.14-9. The data indicate that at water vapor levels as low as 0.032 weight percent in nitrogen trifluoride, there is significant enhancement of corrosion with an alloy such as 316 ELC stainless steel exhibiting the greatest resistance to corrosion of the metal specimens tested. The post-test condition of the metal specimens is shown in Figures 2.14.5 and 2.14.6. The corrosivity enhancement due to the presence of water in the nitrogen trifluoride is very similar to that produced by the presence of hydrogen fluoride. The presence of water vapor in gaseous nitrogen trifluoride should be minimized at all times to levels which are practically attainable.

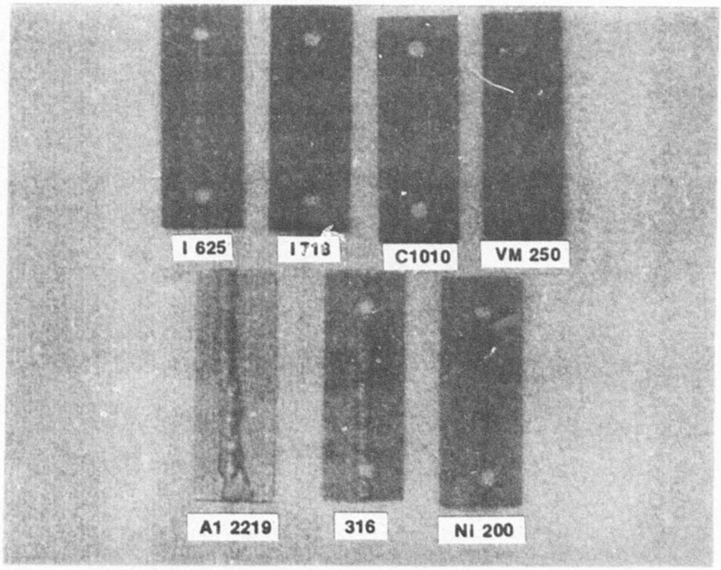


Figure 2.14.5. Metal Specimens After 25 Days Static Exposure to 0.1 Weight Percent Water in Nitrogen Trifluoride at 3.45 MN/m^2 (500 psia) and 344 °K (160°F)

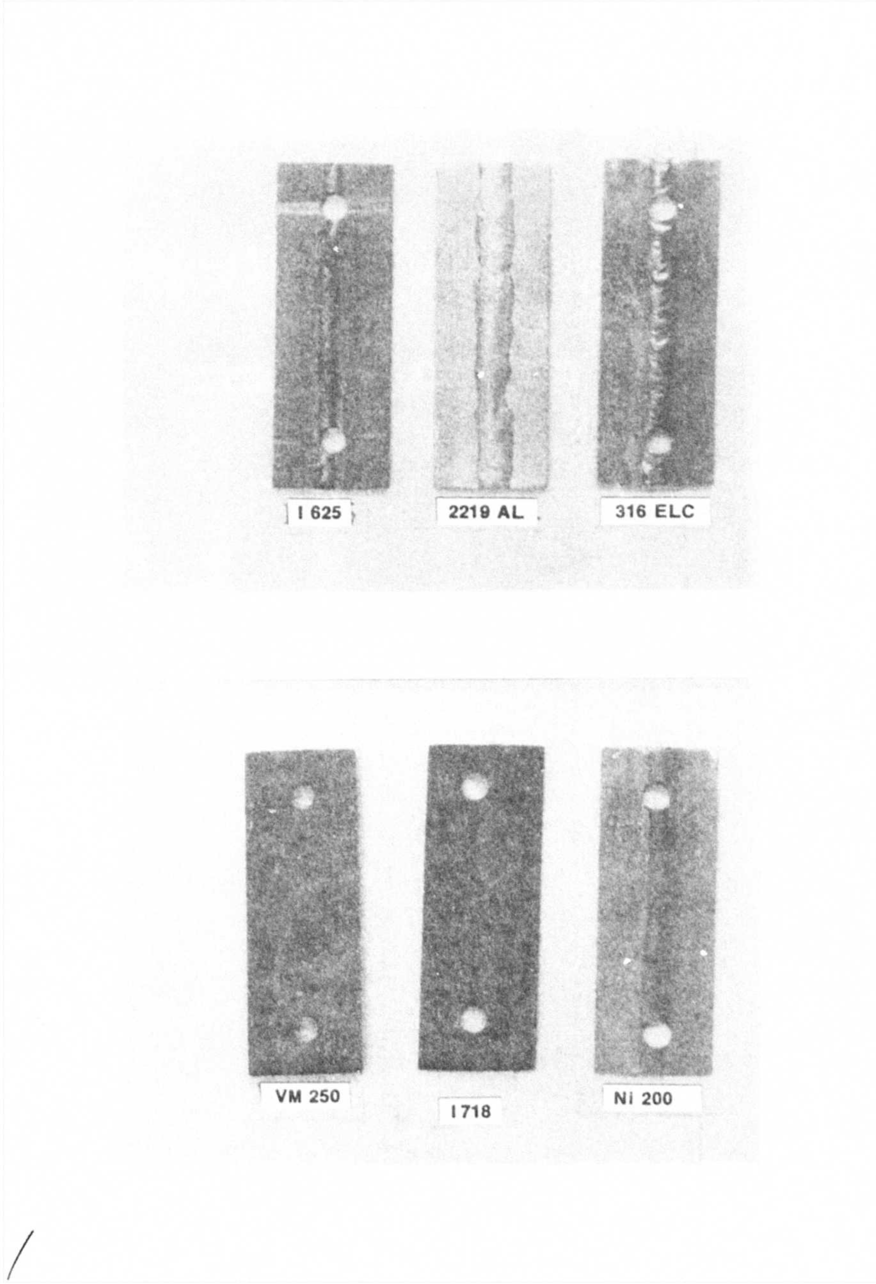


Figure 2.14.6. Metal Specimens After 29 Days Static Exposure to 0.032 Weight Percent Water in Nitrogen Trifluoride at 3.45 MN/m^2 (500 psia) and 344 PK (160°F)

2.0, Experiment Results and Discussion

2.15 GASEOUS CORROSION RATES OF METALS UNDER FLOW CONDITIONS

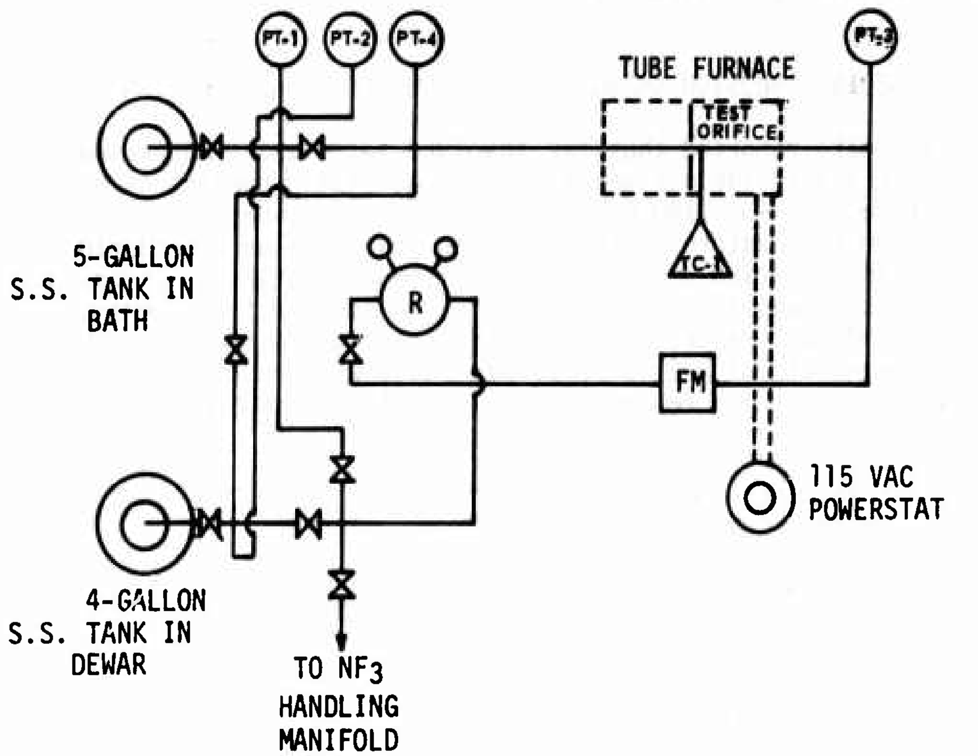
The data reported thus far involved tests which determined the compatibility of NF_3 with various metals either at static conditions at a maximum temperature of 344 K (160 F) or at dynamic conditions of short duration at much higher temperatures. The tests in this task were conducted at 400 K (260 F) for a period of 8 hours under flow conditions. In cases in which the corrosion was nil at 400 K (260 F) no further tests were conducted with the candidate materials. If significant corrosion did occur, the test was repeated at 322 K (120 F) to ascertain whether significant corrosion occurs at the lower temperature. The metal candidates used in this task were:

Nitronic - 40
316 ELC Stainless Steel
Inconel 718
Inconel 625
Aluminum Bronze 623
Narloy A

2.15.1 Apparatus and Procedures

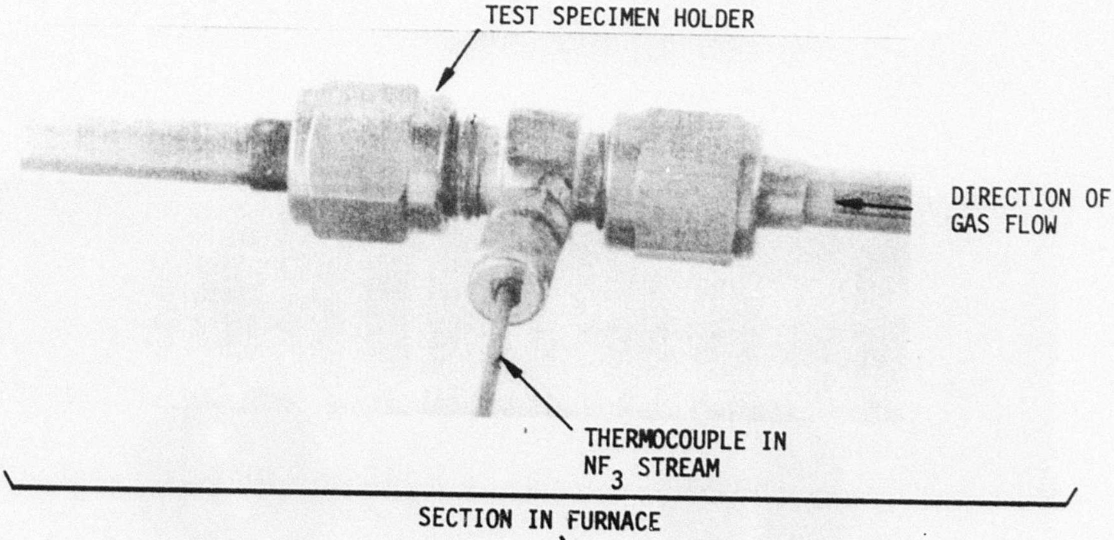
A schematic diagram of the apparatus in which the tests were conducted is shown in Figure 2.15.1; a photograph of the apparatus is shown in Figure 2.15.2; and representative test specimens are shown in Figure 2.15.3. The test specimens were approximately 1.6 mm thick and were drilled to provide a 0.21 mm orifice through which the gaseous nitrogen trifluoride flowed. The gaseous nitrogen trifluoride was condensed in liquid nitrogen after passing through the orifice. A nominal pressure drop of 1.72 MN/m^2 (250 psi) was maintained across the orifice during the entire test duration and the flow rates through the orifices ranged between 6,000 and 8,000 cc/min.

Prior to each test the orifice discs were weighed and photographed at approximately 75-fold magnification. The flowrate through the orifices was monitored periodically during the test; and the flowrate at the end of each test was restored to the initial flowrate value and the pressure drop values were compared to determine if significant changes occurred. The flow meter readings could be read to within 100 cc/min and the repeatability of the flowmeter was within 250 scc/min. The implication of the repeatability value is that under the test conditions used, a change in the pressure drop value of 69 KN/m^2 (10 psi) is within the limits of repeatability of the flow meter.



TRANSDUCER PT-1	0-2000 PSIG RANGE, 5-GALLON S.S. TANK	
TRANSDUCER PT-2	0-2000 PSIG RANGE, 4-GALLON S.S. TANK	
TRANSDUCER PT-3	0- 500 PSIG RANGE, UPSTREAM ORIFICE	
TRANSDUCER PT-4	0- 500 PSIG RANGE, DOWNSTREAM ORIFICE	
TC-1	C-A THERMOCOUPLE, UPSTREAM OF ORIFICE	
R	NF_3 REGULATOR	
FM	MATHESON MASS FLOWMETER	

Figure 2.15.1. Schematic Diagram of Test Apparatus Used in Gaseous Corrosion Tests Under Flow Conditions



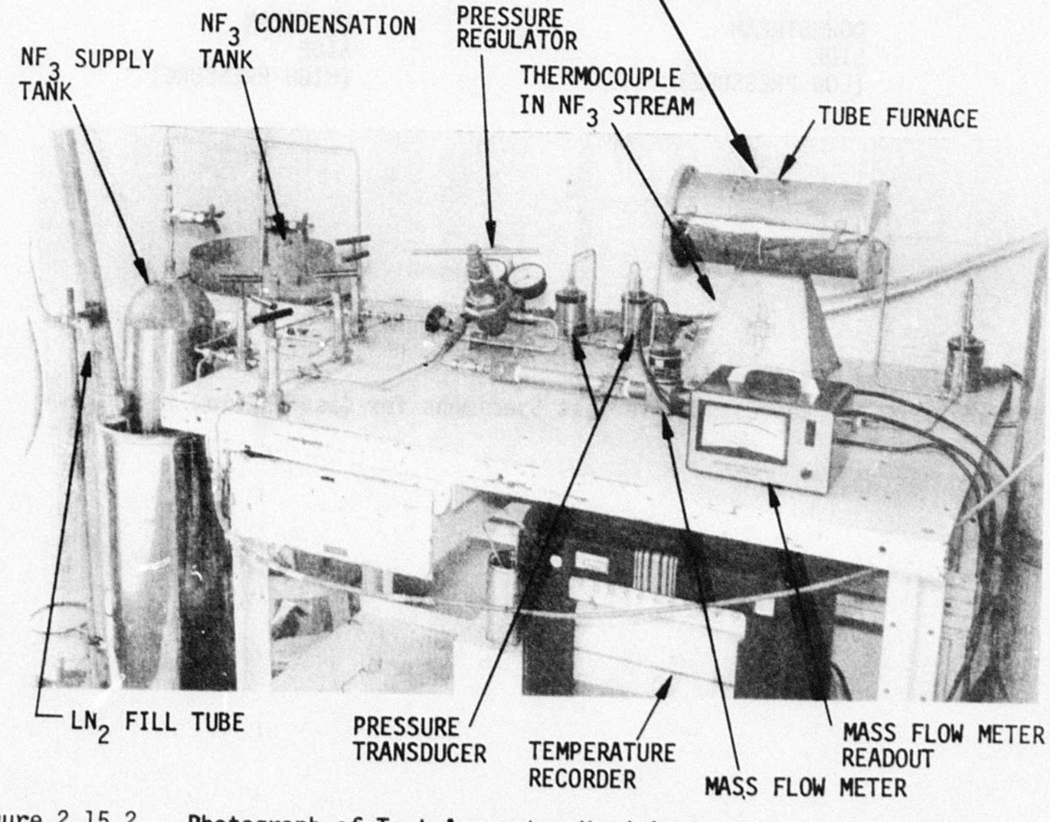
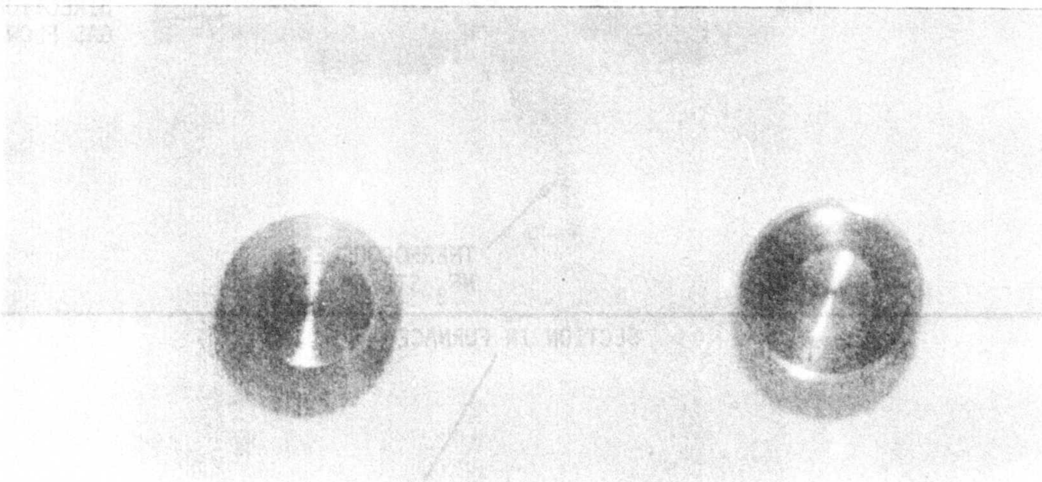


Figure 2.15.2. Photograph of Test Apparatus Used in Gaseous NF_3 Flow Tests



DOWNSTREAM
SIDE
(LOW PRESSURE)

UPSTREAM
SIDE
(HIGH PRESSURE)

Figure 2.15.3. Representative Test Specimens for Gaseous Flow Tests

2.15.2 Test Results

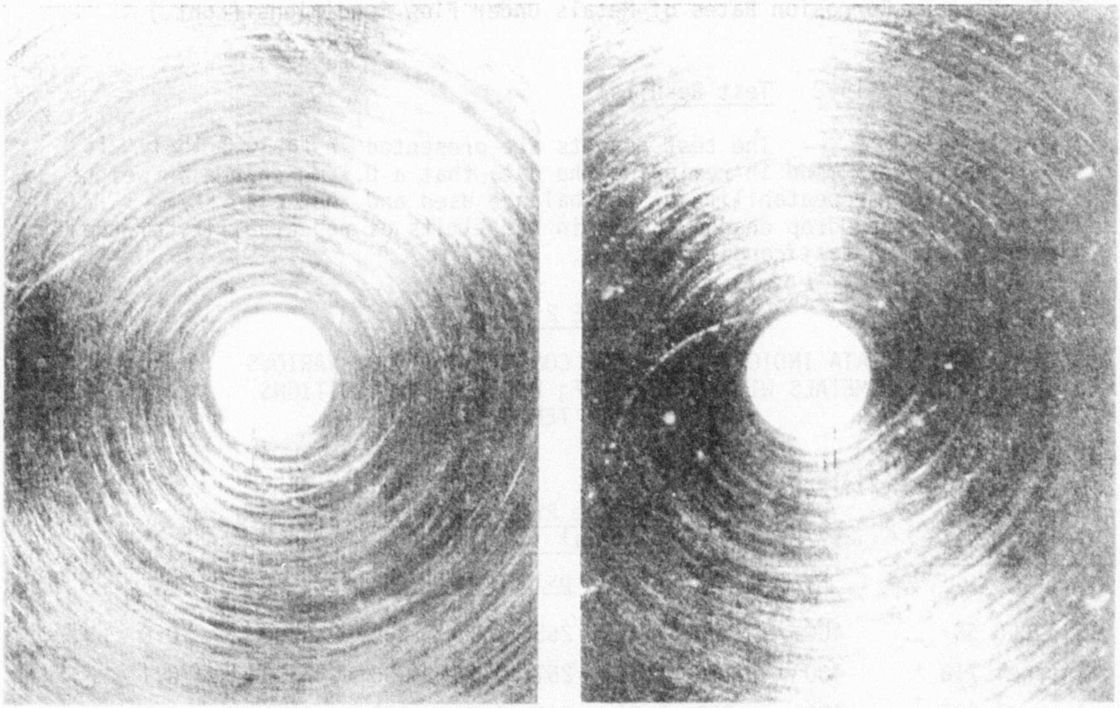
The test results are presented in Table 2.15-1. It should be kept in mind in reviewing the data that a 0.1 mg change in weight is the limit of repeatability of the balance used and that a 69 KN/m^2 (10 psi) pressure drop change is within the limits of repeatability of the flowmeter at the test conditions used.

TABLE 2.15-1

DATA INDICATIVE OF THE COMPATIBILITY OF VARIOUS METALS WITH GASEOUS NF_3 UNDER FLOW CONDITIONS AT MODERATE TEMPERATURES

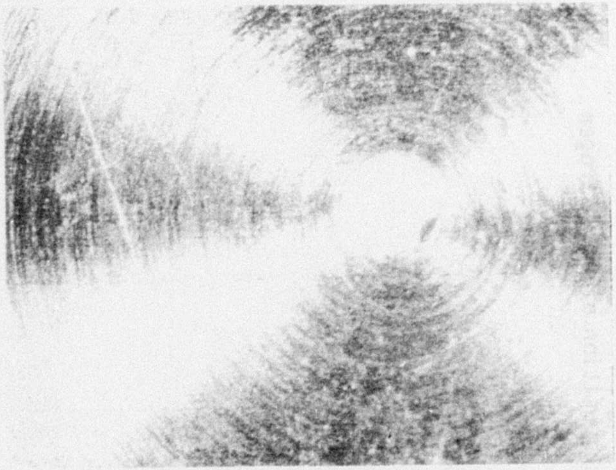
Material	Exposure Temperature		Orifice Pressure Drop				Flowrate scc/min	Weight Change
			Initial		Final			
	K	F	MN/m^2	psi	MN/m^2	psi		mg
316 ELC SS	400	260	1.76	255	1.79	260	6700	0.0
Inconel 718	400	260	1.73	251	1.76	256	7300	0.1
Inconel 625	400	260	1.72	249	1.78	259	6300	0.1
Nitronic 40	400	260	1.72	249	1.78	258	7100	0.4
Aluminum Bronze 623	400	260	1.67	243	1.71	248	7300	0.0
Narloy A	400	260	1.71	248	1.89	274	8200	1.4
Narloy A	322	120	1.71	248	1.78	259	7700	0.1

The significant items to note from the data are that (1) at 400 K (260 F) no significant corrosion of 316 ELC SS, Inconel 718, Inconel 625, or Aluminum Bronze 623 occurred during exposure to flowing, gaseous nitrogen trifluoride for eight hours; (2) at 400 K the Nitronic 40 exhibited no significant change in pressure drop or visual appearance (see Figure 2.15.4), but a 0.4 mg weight loss was detected, the loss was not apparently due to corrosion; (3) at 400 K, the Narloy A specimen did undergo a significant change in pressure drop during the eight hour period, a significant weight loss, and a significant change in appearance; the specimen was a dark red mahogany color when removed from the test fixture and the coloration turned white upon exposure to the atmosphere; some of the corrosion product was readily removed by a water wash and other portions of the product required brushing to achieve removal (see Figure 2.15.5); assuming that



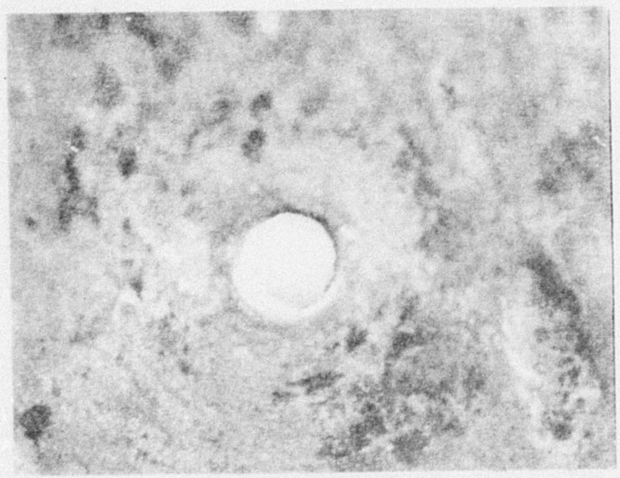
BEFORE EXPOSURE AFTER EXPOSURE

Figure 2.15.4. Upstream Face of Nitronic 40 Test Specimen Before and After Exposure to Gaseous NF_3 at 400K (260F) for 8 Hours at 1.83 MN/m^2 (250 psig) (Magnification ~75X)

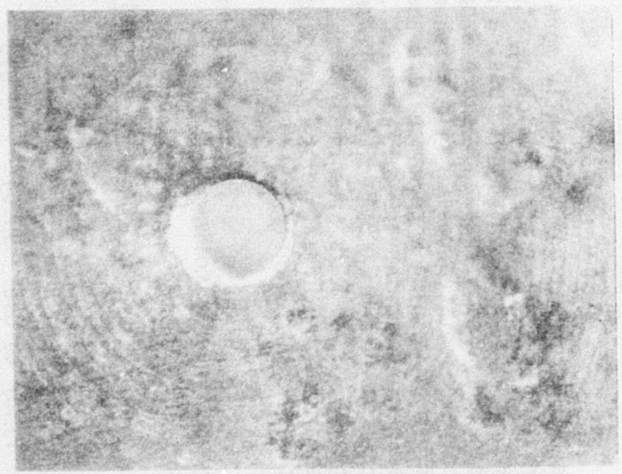


a. BEFORE EXPOSURE TO NF_3

b. AFTER EXPOSURE TO NF_3 FOR 8 HOURS AND TO AIR FOR 1/2 HOUR



c. AFTER EXPOSURE TO NF_3 AND AFTER A WATER WASH



d. AFTER EXPSOURE TO NF_3, WATER WASH AND BRUSHING

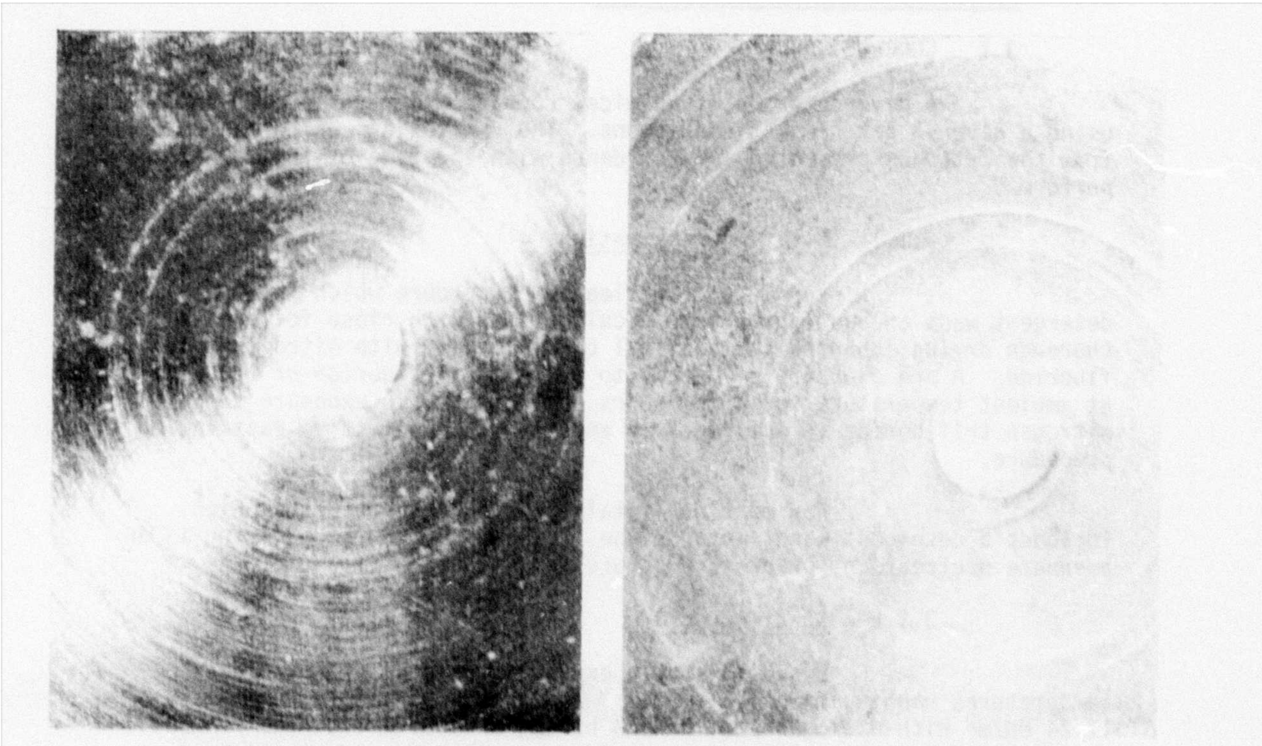
Figure 2.15.5. Surface of Narloy A Specimen Before and After Exposure to Gaseous NF_3 at 400K (260F) for 8 Hours at 1.83 MN/m^2 (250 psig) (Magnification ~75X)

the corrosion was not apparently more severe in the flow channel than on the surface of the disc a corrosion penetration of 188 mpy was calculated for the specimen based on weight loss; a repeat of the test at 322 K (120 F) indicated no significant pressure drop or weight change, but visual inspection revealed a corrosive film which darkened on exposure to air and when removed by washing with water left the surface of the Narloy A in a smoother conditio . (See Figure 2.15.6; note the initial turning marks are much less evident after exposure.) The large penetration rate value for Narloy A at 400 K is predicted by the relatively short exposure period. After formation of a layer of corrosion products, the corrosion rate should decrease significantly.

The composition of the nitrogen trifluoride used in the tests was as follows.

	Composition, Weight Percent						
	NF_3	Active F^- as HF	N_2	CO/O_2	CF_4	CO_2	N_2O
Prior to Use	99.64	0.016	Tr	0.31	.014	Tr	.025
After Use	99.68	0.0016	Tr	0.26	.021	Tr	.033

The data indicate that the no significant decomposition of the nitrogen trifluoride occurred during the testing.



BEFORE EXPOSURE AFTER EXPOSURE

Figure 2.15.6. Surface of Narloy A Specimen Before and After Exposure to Gaseous NF_3 at 322 (120F) for 8 Hours at 1.83 MN/m^2 (250 psig) (Magnification ~75X)

3.0 CONCLUSIONS AND RECOMMENDATIONS

3.1 CONCLUSIONS

A diverse group of chemical compatibility tests were conducted using a diverse set of test conditions. The conclusions which can be drawn from the data are presented in accordance with the type of tests which were performed.

3.1.1 Cleaning-Passivation

For metals, a cleaning procedure which includes a detergent wash and an appropriate pickling step with rinse followed by thorough drying enhances the chemical compatibility with nitrogen trifluoride. A pre-exposure of metals to nitrogen trifluoride or fluorine at ambient temperature for a few hours prior to static exposure to nitrogen trifluoride is not required and is not an effective passivation procedure.

For most non-metals, a cleaning procedure which includes a detergent wash, water rinse, followed by thorough drying is an adequate pretreatment prior to exposure to nitrogen trifluoride.

3.1.2 Static Exposure

Based on static exposure of metals for 270 days at temperatures ranging from 195 to 344 K and pressures ranging from 1.38 to 17.24 MN/m^2 with nitrogen trifluoride having an active fluoride content of 0.1 percent or less, no metal candidate exhibited a corrosion penetration rate greater than 0.35 pm/sec (0.43 mpy). Generally, a corrosion penetration rate of 0.8 pm/sec (1 mpy) or less is considered acceptable for long-term compatibility.

Based on the static exposure of non-metallic materials for 270 days at a temperature of 195 K to either liquid- or vapor-phase nitrogen trifluoride, all the materials appeared to be acceptable except for the greases which were dispersed throughout the system. The elastomers did exhibit degradation in mechanical properties but did not fail entirely. At 344 K and 3.45 MN/m^2, the elastomers failed after 90 days except for the Kalrez which exhibited significant changes in mechanical properties but did maintain its structural integrity. The greases were thoroughly dispersed throughout the system and are unacceptable for this reason. The thermoplastics except for polypropylene exhibited slight mechanical property degradation during 270 days exposure but are suitable for use in nitrogen trifluoride. At 344 K and 17.24 MN/m^2, polytetrafluoroethylene exhibits a 20% decrease in modulus of rigidity as compared to a 55% decrease for Kel-F 81. Thus polytetrafluoroethylene is the preferred material from a compatibility standpoint.

3.1, Conclusions (cont.)

3.1.3 Effect of Impurities

Both hydrogen fluoride and water enhance the corrosion of metals exposed to nitrogen trifluoride and thus both impurities should be reduced to the minimum concentration levels which are practically possible in nitrogen trifluoride.

3.1.4 Effect of Contaminants

The hydrocarbon contaminants in nitrogen trifluoride systems can initiate destructive failures in use-systems. The presence of brazing flux can significantly lower reaction thresholds.

3.1.5 Fracture Mechanics/Toughness

Inconel 718 and 347 stainless steel were not susceptible to stress corrosion cracking in nitrogen trifluoride and C-1018 steel exhibited a very slight susceptibility only in the parent condition.

3.1.6 Gaseous Flow

Nickel 200 was the most corrosion-resistant metal tested at elevated temperatures in flowing gaseous nitrogen trifluoride as evidenced by attaining the highest temperature at which no reaction was apparent. The non-metals tested were all suitable in their normal temperature use range.

3.1.7 Adiabatic Compression

Nickel 200 and 304 stainless steel were found to be the superior metals in resistance to chemical attack during adiabatic compression, while OFHC copper and 6Al-4V titanium were least resistant. Of the non-metals tested, Carbon CJPS rated most resistant to adiabatic compression, while polytetrafluoroethylene, Kel-F 81 and Kalrez were moderately resistant to attack during the adiabatic compression process.

3.1.8 Mechanical Impact

2219 Aluminum is not sensitive to mechanical impact in liquid nitrogen trifluoride at the 11 kg-m energy level; 5Al-2.5 Sn titanium exhibits sensitivity above the 10 kg-m energy level. The threshold levels for reaction of non-metals in liquid nitrogen trifluoride were: greater than 11 kg-m for polytetrafluoroethylene and Kel-F 81, 9.7 kg-m for PFA Teflon, 6.9 kg-m for Viton, Class I. These energy level values are no less than those measured for the same materials in liquid oxygen.

In high pressure gaseous nitrogen trifluoride only non-metallic materials were tested. The threshold energy levels were found to be: 3.45 kg-m at 7.0 MN/m^2 for PFA Teflon, 2.72 kg-m at 7.0 MN/m^2 for Kel-F 81, >11 kg-m at 7.0 MN/m^2, 9.0 kg-m at 8.72 MN/m^2, and 3.45 kg-m at 17.34 MN/m^2 for polytetrafluoroethylene, and >11 kg-m at 17.34 MN/m^2 for Viton, Class I.

3.1.9 Liquid Flow-Impact

The liquid velocity impacting on the heated material surfaces had only a minimal effect on the reaction threshold temperatures. All the non-metals tested were not attacked at their maximum usage temperature. Of the metals tested, Nickel 200 exhibited resistance to attack at the highest temperature, none of the metals were attacked below their maximum usage temperatures.

3.1.10 Waste Disposal

Preheated activated charcoal is a suitable reactant for conversion of nitrogen trifluoride to innocuous and environmentally-acceptable compounds.

3.1.11 Water-Hammer Effects

Of the non-metals tested, Kel-F 81 was superior in resistance to attack by liquid nitrogen trifluoride when subjected to the "water-hammer" effect. Polytetrafluoroethylene was almost as resistant as the Kel-F 81.

3.1.12 Passivation Films

Passivation films on metals are not rapidly formed by nitrogen trifluoride at temperatures from 195 to 344 K, and there is evidence of minimal solubility of the fluorides which are present at the surface of some metals in liquid nitrogen trifluoride.

3.1.13 Gaseous Corrosion Rates

Of the metals tested at 400 K and 1.7 MN/m^2 under flow conditions only Narloy A exhibited significant attack during 8 hours of exposure; 316 ELC stainless steel, Inconel 718, Inconel 625, and Aluminum Bronze 623 were not affected by the nitrogen trifluoride.

3.2 RECOMMENDATIONS

The following recommendations are made for further work.

3.2.1 Additional testing of candidate metals should be conducted under flow conditions at moderate temperatures in gaseous nitrogen trifluoride.

3.2.2 Tests should be conducted to establish suitable valve components and designs for gaseous nitrogen trifluoride usage.

3.2.3 Additional tests should be conducted with elastomers at temperatures of 344 K and lower in hardware assemblies.

3.2.4 The search for and evaluation of suitable lubricants for nitrogen trifluoride systems should be continued.

3.2.5 Physical properties of nitrogen trifluoride which should be experimentally determined are as follows: viscosity of the liquid from 77 to 200 K, densities at 170 to 233.9 K, surface tension at 77 to 144 K, gaseous P-V-T data, adiabatic compressibility/sonic velocity, thermal conductivity of the liquid, heat capacity of the liquid at temperatures greater than 144 K, isothermal compressibility of the liquid, and the dielectric constant of the liquid.

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APPENDIX A

TYPICAL COMPOSITIONS OF CANDIDATE MATERIALS

COMPOSITIONS OF CANDIDATE NON-METALS

ELASTOMERS

Viton - Hydrocarbon cross-linked vinylidene fluoride-hexa fluoropropylene

Silastic LS53 - Fluoroalkyl polysiloxane (fluorosilicone)

Neoprene - polychloroprene

Kalrez (Dupont ECD-006) - copolymer consisting of about 60 mole percent polytetrafluoroethylene, 40 mole percent perfluoro(methylvinyl ether) and 2 mole percent or less of perfluoro(phenylvinyl ether) cross-linked at pendant perfluorophenyl groups with a hydrocarbon bridge.

THERMOPLASTICS

Polyethylene - saturated polymer of ethylene

Polypropylene - saturated polymer of propylene

Tygon - plasticized polyvinylchloride

Mylar - film form of polyethylene terephthalate

Lucite - polymethyl acrylate

Polytetrafluoroethylene - saturated polymer of tetrafluoroethylene

Teflon FEP - polyperfluoropropylene

Teflon PFA - Copolymer of tetrafluoroethylene with perfluoroalkoxy side chains.

Rulon (CaF_2 filled) - Calcium fluoride filled polytetrafluoroethylene

Kel-F 81 - polytrifluoromonochloroethylene

THERMOSETTING POLYMERS

Epoxy EA 934 - believed to be a phenolic resin modified epoxy resin containing asbestos and aluminum filler.

Kevlar - aramid polymer in fiber form.

COMPOSITIONS OF CANDIDATE NON-METALS (cont.)

GRAPHITES

CDJ-83 - medium density bulk graphite impregnated with phosphate salt.

CJPS - medium density bulk graphite impregnated with a special oxidation resistant treatment.

LUBRICANTS

Krytox - polyfluoroalkylester

3L-38RP - vacuum stripped version of polyfluoroalkylester containing low molecular weight, sub-micron size particles of polytetrafluoroethylene.

MS-122 - a low molecular weight, sub-micron particle size form of polytetrafluoroethylene

FS 3451 - a grease consisting of low molecular weight fluoroalkyl polysiloxane thickened with low molecular weight, sub-micron size particles of polytetrafluoroethylene

NOMINAL COMPOSITIONS OF CANDIDATE METALS

STAINLESS STEELS - Fe BASE

Type	C, Max	Mn Max	P Max	S Max	Si Max	Cr	Ni	Others
301	0.15	2.00	0.045	0.030	1.00	16.00 to 18.00	6.00 to 8.00	
303	0.15	2.00	0.20	0.030	1.00	17.00 to 19.00	8.00 to 10.00	
304	0.08	2.00	0.045	0.030	1.00	18.00 to 20.00	8.00 12.00	
304L	0.03	2.00	0.045	0.030	1.00	18.00 to 20.00	8.00 12.00	
316L	0.03	2.00	0.045	0.030	1.00	16.00 to 18.00	10.00 to 14.00	2.00-3.00 Mo
321	0.08	2.00	0.045	0.030	1.00	17.00 to 19.00	9.00 to 12.00	5XC min Ti
347	0.08	2.00	0.045	0.030	1.00	17.00 to 19.00	9.00 13.00	10XC min Cb + Ta
17-4 PH	0.07	1.00	0.04	0.03	1.00	15.50 to 17.50	3.00 to 5.00	Cu 3.00 to 5.00 Cb + Ta 0.15-0.45
A286	0.08	1.00 to 2.00	--	--	0.40 to 1.00	13.50 to 16.00	24.00 27.00	Mo 1.00 to 1.75 Ti 1.90 to 2.30 V 0.10 to 0.50 Al 0.35 Max B 0.003-0.010
Nitronic 40	0.08	8.00 to 10.00	0.060	0.030	1.00	19.25 to 21.50	5.50 7.50	N 0.15-0.40
Carpenter 455	0.03	0.50	0.015	0.015	0.50	11.00 to 12.50	7.50 9.50	Cb + Ta 0.50 Max Ti 0.90 to 1.40 Ca 1.50 to 2.50 Mo 0.50 Max
INCONELS								
625	0.10	0.50	0.015	0.015	0.50	20.00 to 23.00	Base	8.00-10.00 Mo Cb + Ta 3.15-4.15 Al 0.40 Max Ti 0.40 Max Co 1.00 Max Fe 5.0 Max
718 STA	0.08	0.35	0.015	0.015	0.35	17.00 to 21.00	+Co 50.00-55.00	Fe Base Cb + Ta 4.75-5.50 Mo 2.80-3.30 Ti 0.65-1.15 Al 0.20-0.80 Co 1.00 Max B 0.006 Max Cu 0.30 Max
MARAGING STEELS								
200	0.03	0.10	0.01	0.01	0.10	--	18.50	Al 0.10 B 0.003 Co 8.50 Mo 3.25 Ti 0.20 Zr 0.02
250	0.02	0.10	0.010	0.010	0.10	--	18.00 to 19.00	Co 7.00-8.50 Mo 4.60-5.00 Ti 0.30-0.50 Al 0.05-0.15 B 0.006 Max Zr 0.02 Max
LOW CARBON STEELS								
1010	0.08 to 0.13	0.3 to 0.6	0.040	0.050	--	--	--	Remainder Fe
1018	0.15 to 0.20	0.6 to 0.9	0.040	0.050				Remainder Fe
1020	0.18 to 0.23	0.30 to 0.60	0.040	0.050	--	--	--	Remainder Fe

NOMINAL COMPOSITIONS OF CANDIDATE METALS (cont.)

NICKEL

Type	C, Max	Mn Max	P Max	S Max	Si Max	Cr	Ni	Others
200	0.15	0.35	--	0.01	0.35	--	99.0 Min	Cu 0.25 Max Fe 0.40 Max
270	0.02	0.001		0.001	0.001		99.97 Min	Cu 0.001 Max Fe 0.005 Max Co 0.001 Max Co 0.001 Max Mg 0.001 Max Ti 0.001 Max
MONEL								
400	0.30	2.00		0.024	0.50		+Co 63.00- 70.00	Fe 2.50 Max Cu Bal
COPPER ALLOYS								
OFHC CA 101	--	--	0.0003	--				Te .0010 Cu 99.99
Be-Cu CA172								Be 1.8-2.0 Cu + Ag 99.5 Fe+Ni+Co 0.60 Max
Al BRONZE								
CA 623	--	--	--	--	0.25	--	--	Cu 99.5 Fe 2.0-4.0 Sn 0.20 Max Al 8.00-10.00

ALUMINUM ALLOYS

Type	Si Max	Fe Max	Cu Max	Mn	Mg	Cr	Zn Max	Others
1100	1.0	Si+Fe	0.20	0.05	--	--	0.10	
2014	0.5- 1.2	1.0	3.9 to 5.0	0.40 to 1.2	0.20 to 0.8	0.10	0.25	
2219	0.20	0.30	5.8 to 6.8	0.20 to 0.40	0.02	--	0.10	
6061	0.40 to 0.8	0.7	0.15 to 0.40	0.15	0.8 to 1.2	0.04 to 0.35	0.25	

TITANIUM ALLOYS

Type	Al	C Max	H Max	Fe Max	N Max	O Max	V Max	Others
6Al-4V	5.50 to 6.75	0.10	0.0125	0.30	0.05	0.20	4.50	
5Al-2.5 Sn ELI	4.7 to 5.6	0.08	0.0175	0.15	0.05	0.12		Sn 2.00-3.00

TUNGSTEN

	W, Min	Thoria	Other % Max
EW Th-1	98.5	0.8- 1.2	0.5
EW TH-2	97.5	1.7- 2.2	0.5